

Synthesis, Structural Characterization, and Antibacterial Activity of a Novel Quaternized Norbornene Carboxamide against Sulfate-Reducing Bacteria

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Research Article

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Abstract

Microbiologically influenced corrosion associated with sulfate-reducing bacteria (SRB) is a significant concern in oil and gas production and transportation systems due to its contribution to accelerated metal deterioration and hydrogen sulfide generation. This study aimed to synthesize and characterize a novel benzyl bromide complex based on the amide of norborn-5-ene-2-carboxylic acid and diethylenetriamine and to evaluate its antibacterial activity against SRB. The target amide was synthesized by condensation of norborn-5-ene-2-carboxylic acid with diethylenetriamine, followed by quaternization with benzyl bromide. The molecular structure of the synthesized complex was confirmed by Fourier-transform infrared, ¹H and ¹³C nuclear magnetic resonance spectroscopy. Density, refractive index, specific electrical resistance, and electrical conductivity were determined to assess its physicochemical characteristics. Antibacterial activity was evaluated against *Desulfovibrio desulfuricans* in Postgate B medium at inhibitor concentrations of 25, 50, and 75 mg/L. Bactericidal efficiency was determined based on the suppression of hydrogen sulfide production using iodometric titration. The synthesized complex demonstrated pronounced concentration-dependent antibacterial activity. Bactericidal efficiencies of 82.0%, 94.0%, and 99.5% were achieved at 25, 50, and 75 mg/L, respectively. The marked decrease in hydrogen sulfide generation compared with the untreated control confirmed effective suppression of SRB metabolic activity. These findings indicate that the synthesized benzyl bromide complex combines favorable physicochemical characteristics with high antibacterial efficiency and may serve as a promising multifunctional agent for controlling SRB-associated microbiologically influenced corrosion. Further studies should focus on its corrosion inhibition performance, long-term stability, and applicability under field-relevant oil and gas production conditions.

Keywords: norbornene carboxylic acid, amide, benzyl bromide, sulfate-reducing bacteria, biocorrosion

Introduction

Corrosion of carbon steel remains one of the principal factors limiting the long-term reliability and operational safety of petroleum production, transportation, and processing facilities. Throughout their service life, metallic structures are continuously exposed to complex environments containing dissolved salts, carbon dioxide, hydrogen sulfide, water, and diverse microbial populations. The combined action of these chemical and biological factors accelerates material degradation, resulting in localized corrosion, equipment failure, production interruptions, and substantial economic losses. In addition to the direct costs associated with repair and replacement, corrosion-related failures may increase environmental risks and compromise the safe operation of oilfield infrastructure. These challenges have made the development of effective corrosion-control strategies a continuing priority in both corrosion science and petroleum engineering [1–3].

Among the various degradation mechanisms affecting metallic materials in petroleum production systems, microbiologically influenced corrosion (MIC) has emerged as one of the most complex and economically significant forms of corrosion because microbial activity can substantially accelerate electrochemical degradation. Unlike conventional corrosion, which is primarily governed by physicochemical interactions between the metal surface and the surrounding environment, MIC results from the establishment of microbial biofilms that modify the local chemical conditions at the metal–solution interface. Variations in pH, oxygen concentration, redox potential, and the accumulation of corrosive metabolites within these biofilms create localized environments that promote the initiation and propagation of pitting and other forms of localized corrosion. This complex interaction makes microbiologically influenced corrosion one of the most challenging forms of degradation affecting carbon steel equipment in petroleum production systems, where electrochemical and biological processes proceed simultaneously [4–6]. Among the microorganisms involved in MIC, sulfate-reducing bacteria (SRB) are considered the predominant contributors to microbial corrosion in anaerobic petroleum environments. During anaerobic respiration, these bacteria utilize sulfate as a terminal electron acceptor and produce hydrogen sulfide (H₂S), which increases the corrosiveness of production fluids and promotes the formation of iron sulfide deposits on steel surfaces. In addition to sulfide production, growing experimental evidence indicates that certain SRB species are capable of extracellular electron transfer, thereby directly accelerating anodic metal dissolution. The corrosion process is therefore governed by the combined influence of microbial metabolism, biofilm development and localized electrochemical reactions rather than by hydrogen sulfide generation alone. The limited effectiveness of

conventional mechanical protection and routine operational practices in preventing microbial colonization and biofilm formation has stimulated the development of multifunctional chemical agents designed to inhibit microbial activity while simultaneously enhancing corrosion protection [7–16].

Among the available strategies for mitigating corrosion in aggressive industrial environments, organic corrosion inhibitors remain one of the most effective and economically attractive approaches because of their high inhibition efficiency, structural diversity, and the possibility of tailoring their molecular architecture for specific applications. Unlike inorganic inhibitors, organic compounds can be readily modified to optimize their physicochemical properties, adsorption behavior, and compatibility with different corrosive environments. Their protective performance is primarily associated with the presence of heteroatoms, including nitrogen, oxygen, sulfur and phosphorus, together with π -electron systems capable of interacting with vacant d -orbitals of metallic surfaces. These interactions promote the formation of compact adsorbed films that restrict the transport of aggressive species and significantly reduce the corrosion rate. In addition to their chemical composition, the effectiveness of organic inhibitors is strongly influenced by molecular geometry, electronic structure, hydrophobicity and the stability of the adsorbed protective layer under service conditions [10–13].

Within the broad family of organic corrosion inhibitors, nitrogen-containing compounds occupy a prominent position because nitrogen atoms serve as effective adsorption centers through their lone-pair electrons, facilitating strong interactions with metallic surfaces. This class of compounds includes imidazolines, amides, amidoamines and quaternary ammonium derivatives, which have demonstrated high inhibition efficiency and favorable adsorption characteristics in aggressive petroleum environments. Beyond their corrosion-inhibiting properties, many nitrogen-containing compounds also exhibit antimicrobial activity, allowing them to interfere with microbial colonization and biofilm development. Such multifunctional behavior is particularly advantageous in petroleum production systems, where chemical corrosion and microbiologically influenced corrosion frequently occur simultaneously and require integrated mitigation strategies.

Although imidazolines continue to be widely employed because of their well-established inhibition efficiency and strong adsorption characteristics, increasing attention has shifted toward amide- and amidoamine-based derivatives owing to their greater structural flexibility, lower toxicity, and broader opportunities for molecular modification. The introduction of additional amino groups increases the number of potential adsorption sites, whereas quaternization generates positively charged ammonium centers that enhance electrostatic interactions with negatively charged bacterial cell membranes and biofilm matrices. These structural characteristics make quaternized amide derivatives promising multifunctional compounds capable of combining corrosion inhibition with antibacterial activity, thereby increasing their potential for mitigating microbiologically influenced corrosion under oilfield operating conditions. [14–18].

Although substantial advances have been achieved in the development of nitrogen-containing corrosion inhibitors, important scientific and technological challenges remain. Most reported studies have focused on improving corrosion resistance in acidic or chloride-containing media, whereas comparatively few have investigated compounds specifically designed to mitigate microbiologically influenced corrosion caused by sulfate-reducing bacteria. In many cases, corrosion inhibition and antimicrobial performance have been evaluated independently, providing only limited insight into their combined functionality under conditions relevant to petroleum production systems. Benzyl bromide-functionalized norbornene amide derivatives have received comparatively limited attention despite structural features that are expected to enhance both adsorption on metallic surfaces and interactions with bacterial cell membranes. Systematic information describing the relationship between molecular structure, physicochemical characteristics, and antibacterial performance against sulfate-reducing bacteria also remains limited. The absence of comprehensive structure–property–activity correlations restricts the rational design of multifunctional inhibitors capable of simultaneously controlling microbial activity and corrosion in aggressive oilfield environments. These limitations emphasize the need for the development and systematic evaluation of new quaternized amide derivatives with improved multifunctional performance [18–20].

To address the research gap identified above, this study presents the synthesis and comprehensive evaluation of a newly synthesized benzyl bromide-functionalized norbornene amide derivative as a multifunctional candidate for the mitigation of microbiologically influenced corrosion in petroleum production systems. Rather than evaluating corrosion inhibition and antibacterial activity as independent properties, the present investigation combines molecular synthesis, structural characterization, physicochemical analysis and microbiological assessment within a unified experimental framework. This integrated approach enables a more comprehensive evaluation of the relationship between molecular structure, physicochemical characteristics, and antibacterial performance, thereby providing additional insight into the design of multifunctional nitrogen-containing corrosion inhibitors.

The synthesized derivative was characterized by FTIR, ^1H NMR and ^{13}C NMR spectroscopy to confirm its molecular structure, while selected physicochemical properties, including density, refractive index, electrical resistance, and electrical conductivity, were determined to assess characteristics relevant to practical application. Its antibacterial activity was subsequently evaluated against *Desulfovibrio desulfuricans*, one of the principal sulfate-reducing bacteria associated with microbiologically influenced corrosion, by monitoring bacterial growth and hydrogen sulfide production over a range of inhibitor concentrations. The combination of spectroscopic characterization, physicochemical evaluation, and microbiological investigation provides a systematic basis for assessing the multifunctional performance of the synthesized derivative and for guiding the future design of nitrogen-containing corrosion inhibitors for petroleum production systems.

2. Experimental

2.1. Materials

Cyclopentadiene, acrylic acid, diethylenetriamine (DETA), benzyl bromide, and isopropyl alcohol were used as starting materials for the synthesis. All reagents were of analytical grade and were used without further purification. Deionized water was used throughout the study.

2.2. Synthesis of the Norbornene Carboxamide

Norborn-5-ene-2-carboxylic acid was synthesized via the Diels–Alder cycloaddition of cyclopentadiene and acrylic acid according to the previously described method. The resulting acid was subsequently reacted with diethylenetriamine under solvent-free conditions at 130–140 °C for 1.5–2 h. Water generated during the amidation reaction was continuously removed, and the resulting amide, **N-(2-((2-aminoethyl)amino)ethyl)bicyclo[2.2.1]hept-5-ene-2-carboxamide (NDA)**, was obtained in 92.2% yield.

2.3. Synthesis of the Benzyl Bromide Derivative

The quaternary ammonium derivative was prepared by reacting the synthesized amide with benzyl bromide at a molar ratio of 1:2. Briefly, 0.5 mol of the amide was dissolved in 25 mL of isopropyl alcohol, followed by the addition of 1 mol of benzyl bromide. The reaction mixture was maintained at 50–60 °C for 3 h with continuous stirring. After completion of the reaction, the solvent was removed and the resulting benzyl bromide derivative (NDA+C₇H₇Br) was isolated and dried before further analysis.

2.4. Instrumentation

The synthesized compounds were characterized by Fourier-transform infrared (FTIR) spectroscopy together with ¹H and ¹³C nuclear magnetic resonance (NMR) spectroscopy.

FTIR spectra were recorded on a Bruker Alpha FTIR spectrometer equipped with a diamond ATR crystal over the range of 400–4000 cm⁻¹.

¹H and ¹³C NMR spectra were obtained on a Bruker AM-300 spectrometer operating at 300 MHz using CDCl₃, D₂O, or C₆D₆ as solvents. Tetramethylsilane (TMS) was used as the internal chemical shift reference.

Density measurements were performed using an Anton Paar DMA 4500 M density meter. The refractive index (n_D²⁰), specific electrical resistance, and electrical conductivity were measured using an Abbemat 350/500 refractometer.

2.5. Evaluation of Antibacterial Activity

The antibacterial activity of the synthesized compound was evaluated against the sulfate-reducing bacterium *Desulfovibrio desulfuricans* 1143 isolated from the Absheron–Binagadi oilfield in accordance with GOST 39-151-83 [21].

The bacterial strain was cultivated in Postgate B medium under anaerobic conditions at pH 7.0–7.5. Prior to inoculation, the culture medium was sterilized at 120 °C for 30 min and prepared according to the standard procedure. Anaerobic conditions were established by removing dissolved oxygen before inoculation, and all microbiological manipulations were carried out under sterile conditions.

The synthesized compound was added to the culture medium at concentrations of 25, 50, and 75 mg L⁻¹. The inoculated samples were incubated at 30–32 °C for 14 days. A control medium without inhibitor was prepared and incubated under identical conditions.

The inhibitory effect of the synthesized compound was evaluated by monitoring hydrogen sulfide production, black iron sulfide precipitation, and bacterial growth after incubation.

2.6. Determination of Hydrogen Sulfide

Hydrogen sulfide produced during bacterial growth was quantified by iodometric titration according to OST 39-234-89. The bactericidal efficiency (Z) was calculated using the following equation:

$$Z = \frac{C_1 - C_2}{C_1} \cdot 100\%$$

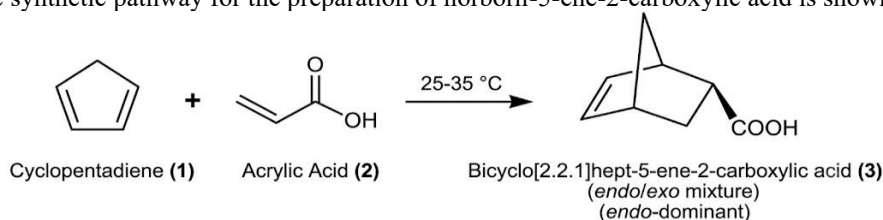
where *C*₁ is the hydrogen sulfide concentration in the inhibitor-free control medium and *C*₂ is the hydrogen sulfide concentration in the inhibitor-treated medium.

Results and Discussion

In the first stage of the synthesis, norborn-5-ene-2-carboxylic acid was prepared via a Diels–Alder cycloaddition reaction between cyclopentadiene and acrylic acid [22]. The reaction was conducted at 25–35 °C and

afforded norborn-5-ene-2-carboxylic acid in 94% yield. The product was obtained predominantly as the endo isomer, accompanied by a minor proportion of the exo isomer.

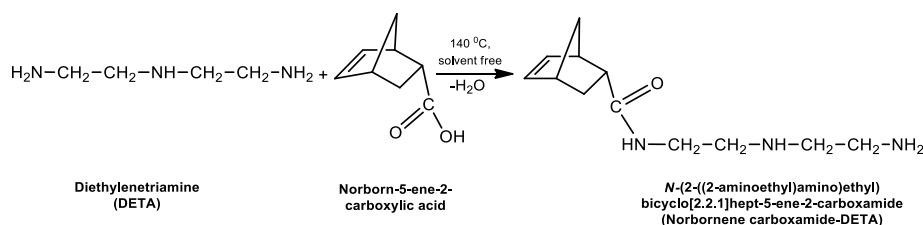
The synthetic pathway for the preparation of norborn-5-ene-2-carboxylic acid is shown in.



Scheme 1. Synthesis of norborn-5-ene-2-carboxylic acid via the Diels–Alder cycloaddition of cyclopentadiene and acrylic acid.

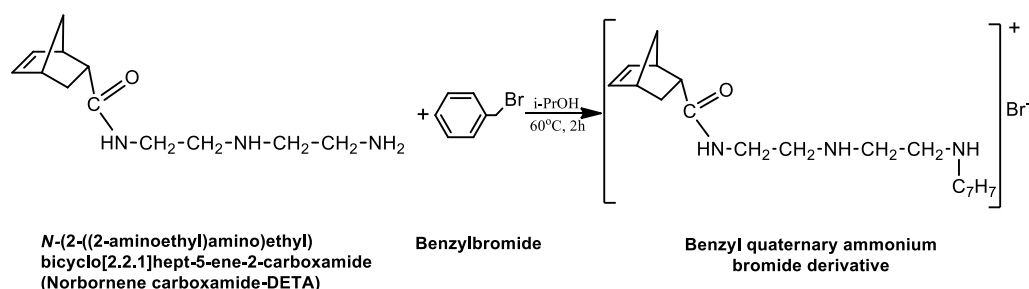
The amide was synthesized by direct amidation of norborn-5-ene-2-carboxylic acid with diethylenetriamine under solvent-free conditions, affording the desired product in 92.2% yield [23]. The reaction was performed at 130–140 °C for 1.5–2 h with the elimination of one mole of water. The resulting amide was isolated as a pale solid and was readily soluble in water and isopropyl alcohol.

The direct amidation proceeded efficiently under the selected reaction conditions, affording the target amide in high yield. The solvent-free procedure provided a straightforward and effective route to the desired intermediate while minimizing the formation of by-products. Introduction of the diethylenetriamine moiety generated both amide and amino functional groups, creating reactive sites suitable for subsequent quaternization. The synthetic pathway is illustrated in Scheme 2.



Scheme 2. Synthetic route for the preparation of N-(2-((2-aminoethyl)amino)ethyl)bicyclo[2.2.1]hept-5-ene-2-carboxamide

The successful quaternization of the amide with benzyl bromide confirms the high reactivity of the amino groups under the selected reaction conditions [24, 25]. The resulting derivative contains permanently charged quaternary ammonium centers, which are characteristic structural features of many antimicrobial compounds. The introduction of these cationic centers increases the ability of the molecule to interact with negatively charged bacterial cell surfaces through electrostatic attraction. In addition, the benzyl substituent increases the hydrophobic character of the molecule, which can promote stronger interactions with the bacterial membrane. The combined effect of the cationic and hydrophobic fragments is likely to contribute to the pronounced antibacterial activity observed for the synthesized compound. (Scheme 3):



Scheme 3. Quaternization of N-(2-((2-aminoethyl)amino)ethyl)bicyclo[2.2.1]hept-5-ene-2-carboxamide with benzyl bromide.

The ¹H and ¹³C NMR spectra of the synthesized amide were recorded and are given in Fig. 1 and Fig. 2. The chemical characteristics of the spectrum are as follows:

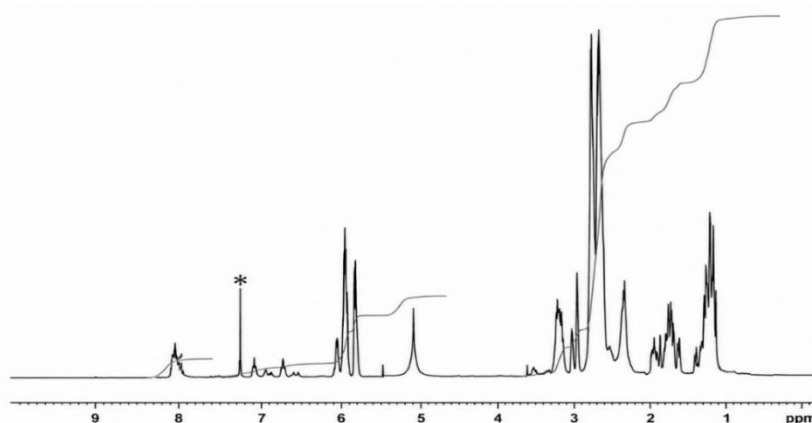


Figure 1. ^1H NMR spectrum of the synthesized N-(2-((2-aminoethyl)amino)ethyl)bicyclo[2.2.1]hept-5-ene-2-carboxamide (NDA).

^1H NMR spectrum, δ , m.p.: 1.21–1.32 m (2H, C^3H), 1.72 d.d.d (2H, C^7H_2 , $J=2.1\text{--}2.2$, 1.2 Hz), 1.93 s (4H, $\text{CH}_2\text{--NH--CH}_2$), 2.64–2.91 m (3H, $\text{C}^{1,2,4}\text{H}$), 3.03–3.24 m (8H, $2\text{N}(\text{CH}_2)_2\text{N}$), 5.1 s (1H, $\text{CH}_2\text{--}\underline{\text{NH}}\text{--CH}_2$), 5.91–5.98 m (1H, $\text{C}^5\text{H=}$), 6.11–6.21 m (1H, C^6H), 8.05 m (1H, CONH).

The ^1H NMR spectrum was in good agreement with the proposed structure of the synthesized compound and confirmed the successful incorporation of the norbornene, amide, and diethylenetriamine fragments. The signal appearing as a multiplet at δ 1.21–1.32 ppm (2H) was assigned to the methylene protons at the C^3 position of the norbornene ring. The resonance at δ 1.72 ppm (2H, $J = 2.1$, 2.2 Hz), observed as a doublet of doublets, was attributed to the C^7 methylene protons. The observed coupling pattern is typical of the constrained bicyclic structure of norbornene, where both vicinal and long-range interactions contribute to signal splitting. A singlet integrating for four protons at δ 1.93 ppm was assigned to the equivalent methylene groups of the $\text{--CH}_2\text{--NH--CH}_2\text{--}$ fragment. The absence of noticeable splitting suggests that these protons experience nearly identical magnetic environments under the experimental conditions. The bridgehead protons of the norbornene framework (C^1 , C^2 and C^4) gave multiplet signals in the δ 2.64–2.91 ppm region. Their relatively downfield position is consistent with the influence of the adjacent carbon–carbon double bond together with the electron-withdrawing character of the neighboring amide group. The resonance between δ 3.03 and 3.24 ppm, corresponding to eight protons, was assigned to the methylene groups of the diethylenetriamine chain. Compared with ordinary aliphatic methylene protons, these signals appear at lower field owing to the deshielding effect exerted by the adjacent nitrogen atoms. The singlet observed at δ 5.10 ppm (^1H) was attributed to the methine proton of the $\text{--CH}_2\text{--NH--CH}_2\text{--}$ fragment adjacent to the amide functionality. Its chemical shift can reasonably be associated with the combined electronic influence of the carbonyl group and possible hydrogen-bonding interactions. Two characteristic olefinic resonances were detected at δ 5.91–5.98 and δ 6.11–6.21 ppm, corresponding to the C^5 and C^6 protons of the norbornene moiety, respectively. The presence of these signals demonstrates that the carbon–carbon double bond remained intact throughout the synthesis. In addition, the amide NH proton appeared as a multiplet at δ 8.05 ppm, which is typical for amide functionalities involved in deshielding interactions caused by the adjacent carbonyl group and hydrogen bonding.

Overall, the ^1H NMR data are consistent with the proposed structure of the synthesized norbornene carboxamide. The characteristic signals corresponding to the norbornene ring, the diethylenetriamine moiety, and the amide group are in agreement with the expected proton environment of the molecule. The presence of the olefinic proton resonances confirms that the carbon–carbon double bond of the norbornene framework was retained during the amidation process. These spectral characteristics are comparable with those reported for structurally related norbornene amide derivatives, supporting the structural assignment of the synthesized compound [26].

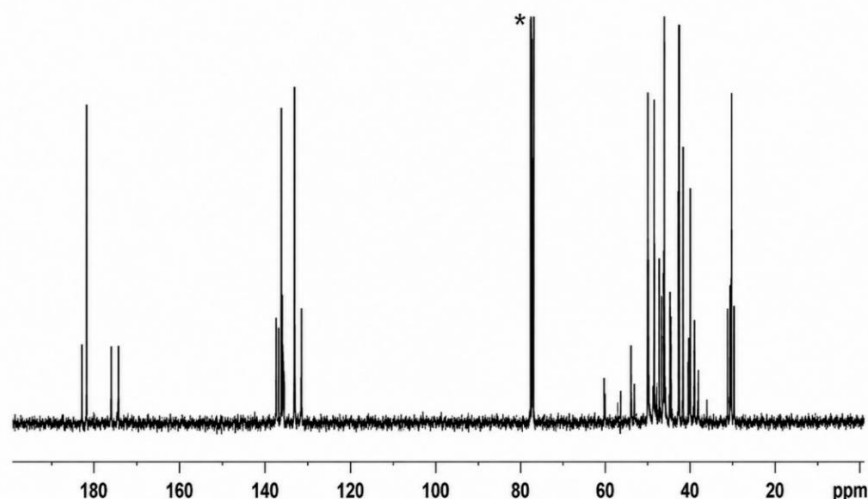


Figure 2. ^{13}C NMR spectrum of the synthesized N-(2-((2-aminoethyl)amino)ethyl)bicyclo[2.2.1]hept-5-ene-2-carboxamide (NDA).

^{13}C NMR spectrum, δ , m.p.: 29.02 (C^3), 38.83 (C^2), 42.01 (C^1), 43.82 (C^4), 45.01 (C^7), 49.02 ($\text{N}(\text{CH}_2)_2\text{N}$), 132 (C^5), 137 (C^6), 178 ($\text{C}=\text{O}$).

The ^{13}C NMR spectrum agreed well with the proposed structure of the synthesized norbornene derivative and enabled the assignment of all carbon atoms in the molecule. The resonance at δ 178 ppm was assigned to the amide carbonyl carbon. Its position in the downfield region is typical of amide functionalities and reflects the reduced electron density around the carbonyl carbon after amide bond formation.

Two resonances observed at δ 132 and 137 ppm were attributed to the olefinic C^5 and C^6 carbons of the norbornene ring. The retention of these signals indicates that the carbon–carbon double bond remained intact during the amidation reaction, showing that the transformation occurred selectively at the carboxyl group while preserving the bicyclic framework.

The saturated carbon atoms of the norbornene skeleton gave signals between δ 29 and 45 ppm. The resonance at δ 29.02 ppm was assigned to the C^3 methylene carbon, whereas the peaks at δ 38.83, 42.01, 43.82 and 45.01 ppm correspond to the C^2 , C^1 , C^4 and C^7 carbons, respectively. The distribution of these resonances reflects the nonequivalent electronic environments of the bridgehead and methylene carbons within the rigid bicyclic structure.

The ^{13}C NMR data are in good agreement with the proposed structure of the synthesized norbornene carboxamide. The presence of characteristic resonances assigned to the amide carbonyl carbon, the olefinic carbons of the norbornene ring, the bicyclic aliphatic framework, and the nitrogen-substituted methylene carbons confirms the successful formation of the target compound. The retention of the olefinic carbon signals further indicates that amidation occurred selectively at the carboxyl group without affecting the carbon–carbon double bond of the norbornene skeleton. The observed carbon chemical shifts are comparable with those reported for structurally related norbornene carboxamide derivatives, providing additional support for the proposed structural assignment.

The physicochemical characteristics of the synthesized $\text{NDA}+\text{C}_7\text{H}_7\text{Br}$ derivative are presented in Table 1. The product was obtained as a dark-brown material with a density of 1.8080 g cm^{-3} and a refractive index of 1.4390. The relatively high density can be attributed to the presence of the bromide-containing quaternary ammonium structure, whereas the measured refractive index reflects the molecular composition of the synthesized derivative. Following quaternization, the compound exhibited a specific electrical conductivity of $5.4 \times 10^{-3}\text{ S cm}^{-1}$ together with a corresponding reduction in specific electrical resistance, indicating the formation of an ionic species. The introduction of quaternary ammonium centers increases the ionic character of the molecule and facilitates charge transport in solution, which is typical of quaternary ammonium salts. These properties may favor interactions between the cationic compound and the negatively charged surface of sulfate-reducing bacterial cells, providing a plausible explanation for the antibacterial activity observed in the subsequent biological evaluation.

Taken together, the measured physicochemical parameters agree well with the proposed structure of the synthesized derivative and provide additional evidence for the successful quaternization of the amide precursor.

The physicochemical properties of the obtained amid-benzyl bromide complex are given in Table 1.

Table 1.

Physico-chemical properties of complex $\text{NDA}+\text{C}_7\text{H}_7\text{Br}$

Reagent	Color	Density, (ρ^{20}_4) g/cm^3	Refractive index, (n^{20}_D)	Specific resistance, ρ , $\text{Om}\cdot\text{m}$	Specific electrical conductivity, σ , S/sm
$\text{NDA}+\text{C}_7\text{H}_7\text{Br}$	dark brown	1.8080	1.4390	$1.85\cdot 10^2$	$5.4\cdot 10^{-3}$

The changes observed in the physicochemical properties after quaternization are consistent with the formation of the proposed quaternary ammonium derivative. In particular, the increase in electrical conductivity together with the decrease in specific electrical resistance reflects the ionic nature of the synthesized compound. These observations complement the spectroscopic characterization, which is discussed in the following section.

The structure of the obtained complex was confirmed by FTIR spectroscopy (Fig. 3) and the following absorption bands were observed:

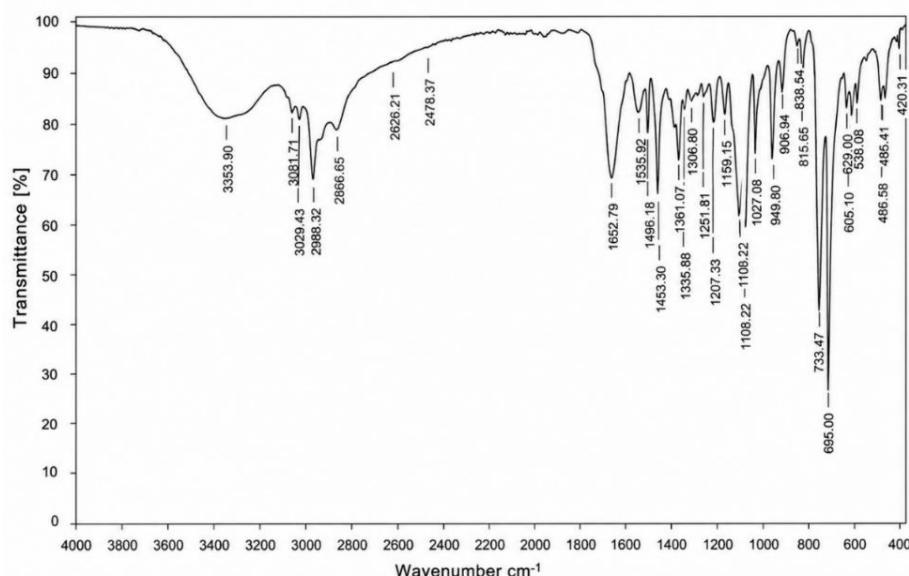


Figure 3. FT IR-spectra of complex NDA+C₇H₇Br

The FTIR spectrum of the synthesized NDA+C₇H₇Br derivative (Figure 3) exhibited the characteristic absorption bands expected for the proposed molecular structure, confirming the successful formation of the target compound. The absorption bands at 695 and 733 cm⁻¹ were assigned to the out-of-plane bending vibrations of the olefinic =C–H bonds of the norbornene ring. Additional bands observed at 815, 906, and 949 cm⁻¹ were likewise attributed to deformation vibrations of the olefinic =C–H groups.

Several absorption bands located at 1027, 1069, 1092, 1159, 1207 and 1251 cm correspond to C–N stretching vibrations, indicating the presence of the amino-containing fragment within the molecular framework. The bands at 1306, 1355, 1361, 1453 and 1496 cm⁻¹ were assigned to the bending vibrations of the aliphatic CH₂ groups, which are consistent with the diethylenetriamine moiety incorporated into the molecule.

The strong absorption band at 1652 cm⁻¹ is characteristic of the amide I band and is attributed to the stretching vibration of the amide carbonyl (C=O) group. Another prominent band at 1535 cm⁻¹ corresponds to the amide II vibration, arising predominantly from N–H bending coupled with C–N stretching. The simultaneous presence of these two characteristic amide bands confirms the formation of the amide functionality in the synthesized compound.

Characteristic absorptions at 2478 and 2626 cm⁻¹ were assigned to the protonated ammonium (NH⁺) group, indicating the formation of the corresponding ammonium bromide derivative after reaction with benzyl bromide. The broad absorption band centered at 3353 cm⁻¹ was attributed to the N–H stretching vibration of amino groups. In addition, the absorption bands observed at 3029 and 3061 cm⁻¹ were assigned to stretching vibrations of the olefinic =C–H bonds associated with the norbornene fragment. The combination of the characteristic ammonium absorptions together with the amide I and amide II bands, as well as the absorptions corresponding to the norbornene and amino fragments, is consistent with the proposed structure of the synthesized NDA+C₇H₇Br derivative.

The bactericidal-inhibitory properties of the received complex amid-benzyl bromide on the basis of norborn-5-ene-2-carboxylic acid and diethylenetriamine were determined. The bactericidal results of the studied complex are shown in the table 2.

Table 2. Effect of the synthesized NDA+C₇H₇Br derivative on the growth of sulfate-reducing bacteria and hydrogen sulfide production.

Complexes conventional name and composition	Concentration of compound, C-mg/l	Amount of bacteria (number of cell/ml)	Amount of H ₂ S, mg/l	Bakterisid al effekt, Z-%
	25	10 ²	68	82

NDA+C ₇ H ₇ Br	50	10 ¹	23.4	94
	75	10 ¹	1.8	99.5
Control-I Amount of H ₂ S in SRB-free medium		24 mg/l		
Control-II Amount of H ₂ S in an environment with SRB		375 mg/l		
Control-III- Number of bacteria in the nutrient medium		10 ⁸ number of cell/ml		

Control I and Control II represent the sterile (bacteria-free) and bacteria-containing control media used to determine the concentration of H₂S. (Control I: H₂S concentration in the SRB-free medium = 24 mg/l; Medium II: H₂S concentration in the SRB-containing medium = 375 mg/l).

Based on the test results, the obtained NDA+C₇H₇Br complex exhibited bactericidal activity of 82% at a concentration of 25 mg/l, 94% at 50 mg/l and 99.5% at 75 mg/l demonstrating a strong inhibitory effect on bacterial growth.

The number of bacteria in the inhibitor-free medium ($n_b \approx 10^8$) is in the hundreds of millions. From this, it can be concluded that one hundred million bacterial cells (10^8) developed in the reagent-free medium. In the samples taken at concentrations of 25, 50 and 75 mg/l in the reagent-containing medium, the number of bacteria changed as follows: at a concentration of 25 mg/l, the number of bacteria decreased from 10^8 to 10^2 , that is, the number of bacteria decreased from one hundred million to one hundred, at concentrations of 50 mg/l and 75 mg/l, it decreased from 10^8 to 10^1 , from one hundred million to ten, thus the bacterial growth is stopped.

Conclusion

1. An amide-benzyl bromide complex based on norborn-5-ene-2-carboxylic acid and diethylenetriamine (NDA+C₇H₇Br) was successfully synthesized, and its bactericidal and inhibitory properties were determined.
2. The obtained complex exhibited bactericidal activity of 82% at a concentration of 25 mg/l, 94% at 50 mg/l and 99.5% at 75 mg/l, demonstrating a strong inhibitory effect on bacterial growth.

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Data Availability

The authors have no competing interests to declare that are relevant to the content of this article. The data that support the findings of this study are available from the corresponding authors upon reasonable request

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Author Contributions

The manuscript was written through contributions of all authors. All authors have given approval to the final version of the manuscript. **CRedit: Babayeva Vafa Hidayat** – conceptualization, data curation, formal analysis, investigation, methodology, validation, visualization, writing-review and editing, **Abbasova Xayala Adil** – formal analysis, **Agamaliyeva Durna Babak** – methodology, formal analysis, **Azizbayli Afaq Rauf** – visualization, writing-review and editing, **Mammadova Nahida Mubariz** – visualization, **Afandiyeva Lala Mahammad** – investigation, **Hasanova Kamila Firudin** – visualization, **İsmaylova Samira Vagif** – data curation, **Afandiyeva Kamala Musa** – visualization, **Mammadova Narmin Marif** – formal analysis, writing-review and editing.

Conflicts of Interest

The authors declare no conflict of interest.

References

1. Soomro, AA, Mokhtar, AA, Kurnia, JC, Lashari, NLU, Sambo, HC. (2022) Integrity assessment of corroded oil and gas pipelines using machine learning: A systematic review. *Engineering Failure Analysis* 131:105810. <https://doi.org/10.1016/j.engfailanal.2021.105810>
2. Vala, M, Kríz, P, Kubes, J. (2024) Addressing hydrogen sulfide corrosion in oil and gas industries: A sustainable perspective. *Sustainability* 16(4):1661. <https://doi.org/10.3390/su16041661>
3. Kumar, AS, Sivakumar, L, Rajadesingu, SS, Malik, T, Parthipan, P. (2025) Sustainable corrosion inhibition approaches for the mitigation of microbiologically influenced corrosion: A systematic review. *Frontiers in Materials* 12. <https://doi.org/10.3389/fmats.2025.1545245>
4. Chang, W, Qian, H, Li, Z, Mol, A, Zhang, D. (2024) Application and prospect of localized electrochemical techniques for microbiologically influenced corrosion: A review. *Corrosion Science* 236:112246. <https://doi.org/10.1016/j.corsci.2024.112246>
5. Dahal, H, Paswan, AM, Dey, J, Raj, CR. (2025) Harnessing the Potential of Amide-Linked Gemini Surfactants for Corrosion Inhibition and Antimicrobial Activity: From Molecular Design to Functional Performance. *Langmuir* 41(8):5455–5466. <https://doi.org/10.1021/acs.langmuir.4c05064>
6. Zamindar, S, Mandal, S, Murmu, M, Banerjee, P. (2024) Unveiling the future of steel corrosion inhibition with special emphasis on N⁺-containing ionic liquids. *Materials Advances*, 5:4563–4600. <https://doi.org/10.1039/D4MA00156G>
7. Talha, M. (2025) Biomacromolecules as green corrosion inhibitors: A review based on mild steel corrosion in acidic media. *Corrosion Reviews*, 43(4): 409–428. <https://doi.org/10.1515/corrrev-2024-0067>
8. Shi, C, Deng, S, Tang, M, Lei, R., Li, X. (2025) Inhibition action and mechanism of the quaternary ammonium cationic surfactant of benzyltrimethylstearyl ammonium chloride on steel corrosion in HCl solution. *Journal of Molecular Structure*, 1337:142170. <https://doi.org/10.1016/j.molstruc.2025.142170>
9. Robles, CE, Olivares-Xometl, O, Lijanová, LV, Likhanova, NV, Lozada, PA, Calvillo-Muñoz, EY, Díaz-Jiménez, V. (2025) Quaternary-ammonium-structure-based ionic liquids as organic corrosion inhibitors of API 5L X52 steel in HCl medium. *Journal of Molecular Structure* 1320:139596. <https://doi.org/10.1016/j.molstruc.2024.139596>
10. Bai, R, Huang, W, et al. (2025) Study on the Synthesis of a New Mannich Base Quaternary Ammonium Salt and Its Synergistic Corrosion Inhibition Performance in a High-Temperature Acidic Environment. *Langmuir*, 41(45):30759–30773. <https://doi.org/10.1021/acs.langmuir.5c04772>
11. Li, J, Chen, L, Wei, B, Xu, J, Wei, B, Sun, C. (2024) Microbiologically influenced corrosion of circulating cooling systems in power plants: A review. *Arabian Journal of Chemistry*, 17(2):105529. <https://doi.org/10.1016/j.arabjc.2023.105529>
12. Rao, P, Mulky, L. (2023) Microbially Influenced Corrosion and its Control Measures: A Critical Review. *Journal of Bio- and Tribo-Corrosion* 9:57. <https://doi.org/10.1007/s40735-023-00772-7>
13. Sun, Y, et al. (2024) Carbon Steel Corrosion Induced by Sulfate-Reducing Bacteria: A Review of Electrochemical Mechanisms and Pathways in Biofilms. *Coatings* 14(9):1105. <https://doi.org/10.3390/coatings14091105>
14. Liu, W, Bi, W, Hu, Y, Lu, W, Feng, W, Wang, Y, Li, Y, Liu, J. (2024) Influence of initial pH and sulfate-reducing bacteria concentration on the microbiologically influenced corrosion of buried pipeline steel. *Materials and Corrosion* 75:1193–1203. <https://doi.org/10.1002/maco.202414320>
15. Hakkarainen, TJ, et al. (2023) Microbiologically influenced corrosion—more than just microorganisms. *FEMS Microbiology Reviews* 47(5) fuad041. <https://doi.org/10.1093/femsre/fuad041>

16. Kumar, AS, Sivakumar, L, Rajadesingu, SS, Malik, T, Parthipan, P. (2025) Sustainable corrosion inhibition approaches for the mitigation of microbiologically influenced corrosion: A systematic review. *Frontiers in Materials* 12. <https://doi.org/10.3389/fmats.2025.1545245>
17. Xueqing Lv, Can W, Jia L., Wolfgang S, Ini-Ibehe NE, Yimeng Z, Ailing X, Jizhou D and Ruiyong Z. (2024) The Microbiologically Influenced Corrosion and Protection of Pipelines: A Detailed Review. *Materials*, 17(20):4996. <https://doi.org/10.3390/ma17204996>
18. Dahal, H, Paswan, AM, Dey, J, Raj, CR (2025) Harnessing the Potential of Amide-Linked Gemini Surfactants for Corrosion Inhibition and Antimicrobial Activity: From Molecular Design to Functional Performance. *Langmuir*, 41(8):5455–5466
19. Zamindar, S, Mandal, S, Murmu, M, Banerjee, P. (2024) Unveiling the future of steel corrosion inhibition with special emphasis on N⁺-containing ionic liquids. *Materials Advances*, 5:4563–4600
20. Shi, C, Deng, S, Tang, M, Lei, R, Li, X. (2025) Inhibition action and mechanism of the quaternary ammonium cationic surfactant on steel corrosion in hydrochloric acid solution. *Journal of Molecular Structure*, 1337:142170
21. Mammadbayli, EH, Babayeva, VH, Aghamaliyeva, DB, Mammadova, NM (2025) Study of bactericide-inhibitor properties against microbiological corrosion process of bromide complex of imidazoline based on norbornene carboxylic acid and diethylenetriamine. *Chemical Problems*. 4 (23):573-579. <https://chemprob.org/wp-content/uploads/2025/10/573-579.pdf> DOI: 10.32737/2221-8688-2025-4-573-579
22. Mammadbayli, EH, Babayeva, V.H. (2024) Investigation of fungicidal properties of hexyl bromide complex of n'-(2,2-bicyclo[2.2.1]hept-5-en-2-yl-4,5-dihydro-1-h-imidazolin-1-ylethyl)ethane-1,2-diamine as antimicrobial additive to lubricant-coolant fluids. *Chemical Problems*. 2(22):168-176. DOI: 10.32737/2221-8688-2024-2-168-176
23. Mammadbayli, EH, Babayeva, VH, Agamaliyeva, DB (2025) Study of the bactericidal properties of allyl bromide complex of amide obtained from norbornene carboxylic acid against biocorrosion. *PPOR*. 26 (1):144-153. <https://doi.org/10.62972/1726-4685.2025.1.144>
24. Mammadbayli, EH, Babayeva, VH, Salamova, NV (2025) Study of surfactant properties of hexylbromide complex of imidazoline based on 5-norbornene-2-carboxylic acid and diethylenetriamine. *Springer Nature Link*. 2:323-332. https://doi.org/10.1007/978-3-031-81564-5_39
25. Abbasov, VM, Babayeva, VH, Mammadbayli, EH, Mammadova, NM, Azizbeyli, AR. (2026) Study of the Inhibitory Properties of the Imidazoline Bromide Complex Based on Norborn-5-Ene-2-Carboxylic Acid and Diethylenetriamine Against H₂S Corrosion. *Springer, Cham*. 1805:539-545. https://doi.org/10.1007/978-3-032-15926-7_58
26. Mammadbayli, EH, Babayeva, VH, Fataliyeva, ZG, Akhmedov, FI, Salmanova, ChG, Azizbeyli, EI. (2025) Study of the bromide complex of amide of norbornene carboxylic acid as a surface-active compound. *PPOR*. 26 (4):1098-1104. <https://doi.org/10.62972/1726-4685.2024.3.704>