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Experimental investigation of sulfur compound elimination from corrosion inhibitors

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Summary:

Corrosion poses significant economic, safety, and environmental challenges in the oil and gas industry, necessitating effective corrosion inhibition strategies. Chemical inhibitors offer a cost-effective solution, yet selecting the appropriate formulation remains complex due to diverse field compositions and metallurgies. Film-forming inhibitors such as phosphate esters, imidazolines, and quaternary ammonium compounds along with sulfur compounds are widely used to combat CO_2 and H_2S corrosion. However, the environmental and health impacts of using sulfur compounds warrant investigation into alternative formulations.

This study aims to assess the synergistic effect of H_2S in the absence of sulfur compounds on C1018 carbon steel corrosion. The corrosion behavior of C1018 carbon steel in 3% NaCl brine with either pure CO_2 or CO_2/H_2S was evaluated using the static bubble test and dynamic rotating cage autoclave experiment at 60 °C and 20 °C to investigate the synergistic effect of H_2S on carbon steel pipes. The influence of pH, temperature, and H_2S concentration on corrosion inhibitor performance was reviewed. Results indicate that phosphate esters, imidazoline, and quaternary amines exhibit higher inhibition efficiency in CO_2/H_2S environments without sulfur compounds. Notably, even at lower H_2S concentrations, imidazolines, and quaternary amines demonstrate stable inhibitory rates. In a field case study, the corrosion rate of an imidazoline corrosion inhibitor was measured. When exposed to 0.035% H_2S, the average corrosion rate was determined to be 0.0365 mm/y. This rate was lower than the corrosion rate observed when both a sulfur compound and 0.035% H_2S were present, which was 0.08 mm/y. Eliminating sulfur compounds from corrosion inhibitor formulations offers environmental benefits and mitigates health risks. The findings underscore the importance of understanding chemistry and complex chemical interactions in corrosion environments and should be investigated further to optimize the inhibitor formulations for enhanced performance and reduced environmental impact.

Preface

This thesis represents the result of extensive academic research and collaboration aimed at increasing knowledge and finding solutions to the essential challenges of corrosion mitigation in the oil and gas sector. This research project was conducted as part of the Master's program in Energy and Environmental Technology at the University of South-Eastern Norway (USN). It was made possible through a successful collaboration with SLB whose advanced laboratory facilities played a crucial role in conducting experimental investigations.

Corrosion is a ubiquitous deteriorating process in the oil and gas industry, with significant effects on the environment, economy, and safety. Film-forming corrosion inhibitors are most commonly used in addressing the corrosive effects of CO_2 and H_2S. Nevertheless, the traditional dependence on sulfur compounds in inhibitor formulations is being closely examined because of environmental and health issues. This calls for the development of new and creative methods for corrosion prevention. The main goal of this project is to investigate the synergy effect of H_2S in the corrosion inhibition mechanism in the absence of sulfur compounds. The provided findings are expected to not only contribute to scientific knowledge but also provide practical ideas for improving the integrity and sustainability of oil and gas infrastructure. All the test and experimental data can be made available.

I would like to express my sincere gratitude to my advisor, Per Morten Hansen, for his invaluable support and guidance throughout the course of this project. I am also thankful to Tore Nordvik, Oystein Birketveit, and Rune Evjenth from SLB for their expertise and guidance during my experiments. They have always been keen to help me navigate experiments and answer questions. Lastly, I would like to thank my colleague Kristina Margareta Fosshagen for teaching me how to operate and troubleshoot the corrosion testing and monitoring methods.

Porsgrunn, 15th May 2024

Aditi Santosh Mhatre

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Nomenclature

A – Area

ASTM – American Society for Testing and Materials

B – Stern-Geary Constant

Ca – Calcium

CO_2 – Carbon-dioxide

DTDPA – Dithiodipropionic acid

FAC – Flow-accelerated Corrosion

Fe – Iron

FeS – Ferrous Sulfide

FFCI – Film Forming Corrosion Inhibitor

H_2CO_3 – Carbonic Acid

H_2S – Hydrogen Sulfide

LPR – Linear Polarization Resistance

ME – 2-Mercaptoethanol

MSDS – Material Safety Data Sheet

N – Nitrogen

OCP – Open Circuit Potential

O – Oxygen

P – Phosphorous

PPE – Personal protective equipment

Ppm – Parts per million

RCA – Rotating Cage Autoclave

Rp – Polarization Resistance

S - Sulfur

SC – Sulfur Compound

SEM – Scanning Electron Microscopy

TGA – Thioglycolic acid

TS – Thiosulphate

VSI – Vertical Scanning Interferometer

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1 Introduction

Corrosion is the predominant deteriorating mechanism in the oil and gas industry, and its importance arises from its significant economic, safety, and environmental ramifications. The occurrence of corrosion-induced failures in critical equipment, such as pressure vessels, boilers, and structural components, presents significant safety concerns due to the potential for catastrophic damage to operations and harm to workers. Corrosion can arise at several points along the hydrocarbon path, including production tubing, downhole valves, wellheads, surface facilities, pipelines, and processing plants. Consequently, it is responsible for the majority of failures in pressure equipment [1], [2]. The continued development of fields in deeper offshore wells with increased temperatures, pressures, and concentrations of carbon dioxide, hydrogen sulfide, and chloride is driving up the cost of corrosion [3]. Economically, corrosion is expected to damage a fourth of the world's yearly steel production, which amounts to around 150 million tons per year. According to the records, the economic expenses of corrosion in natural gas processing and oil refining plants range from 10 to 30% of the maintenance budget [2].

Chemical inhibition via the injection of inhibitors presents a cost-effective approach to safeguarding facilities against internal corrosion. A variety of chemicals have been documented in the literature for controlling corrosion; however, selecting the appropriate formulation remains challenging due to the diverse compositions of fluids and complexities of corrosion sources within a single field. Notably, the effectiveness of an inhibitor in one well may not extend to others within the same field. Considerations for inhibitor selection include the materials to be protected, fluid flow characteristics, and effluent nature. Film-forming inhibitors are particularly valuable for preventing CO_2 and H_2S corrosion, which are prevalent in the oil and gas industries [2].

Corrosion inhibitors are chemical substances that are introduced in low concentrations to a corrosive environment to prevent or slow corrosion without causing substantial reactions with other environmental components. Concentration might range from 1 to 15000 ppm. The addition of corrosion inhibitors to the system slows the corrosion process or reduces the oxidation rate of the metal. Inhibition is the method of preventing the attack of corrosive species by adsorbing inhibitors on a whole metallic substrate [4]

Corrosion inhibitors are classified as follows: passivating(anodic), cathodic, vapor phase, and film forming. Film-forming corrosion inhibitors are commonly used to protect the anaerobic oil, condensate, and gas production systems, sometimes with synergists. They create a protective film on metal surfaces, substantially curbing corrosion rates and preserving the integrity of metal and alloy surfaces [5]. Film forming inhibitors lower the cathodic beta value while the anodic value remains essentially unchanged. These film-forming corrosion inhibitors are useful for preventing chloride, H_2S, and CO_2 corrosion. They can be used for continuous injection or batch treatment, both downhole and at the wellhead [6].

Corrosion inhibitors which are generally used in the oil and gas industries are classified as follows [7]:

- Amides/Imidazoline
- Salts of nitrogen-containing molecules with carboxylic acids
- Nitrogen quaternary salts
- Amines, amides and polyoxyalkylized imidazoline
- Heterocyclic compounds containing N, P, S, and O

Organic compounds containing sulfur heteroatoms have been shown to effectively reduce corrosion. The existence of a single pair of electrons and pi electrons in sulfur-containing molecules enables for easier deposition on the metal surface, increasing their adsorption tendency [8], [9]. Sulfur-containing compounds, such as thiourea and mercaptoethanol, can reduce corrosion on carbon steel in certain conditions. The synergistic corrosion prevention mechanism of mixed inhibitors, including sulfur-containing compounds, has been elucidated, indicating the efficiency of sulfur compounds in producing a protective adsorption layer on the metal surface [10]. However, the role of sulfur compounds in the field conditions is yet unknown.

1.1 Objective

Film-forming corrosion inhibitors may exhibit varying degrees of corrosion protection in real-world conditions compared to controlled laboratory tests. As a result, in addition to the filming agents and the solvent package, a sulfur-containing ingredient is frequently added to the corrosion inhibitor formulation to enhance surfactant adhesion to metal surfaces. Nonetheless, the influence of sulfur ingredients on the performance of corrosion inhibitors under actual field conditions remains a subject of ongoing investigation. Some filming corrosion inhibitors were observed to work successfully without sulfur compound in the field. But when the same corrosion inhibitor was tested in the laboratory setup, it did not perform [11]. Furthermore, it is imperative to acknowledge that the conventional sulfur compounds commonly employed in the oil and gas industry raise significant environmental concerns. Their use poses potential health risks to workers and nearby communities, and their disposal and transportation involve substantial costs [12], [13].

The purpose of this study is to eliminate the sulfur compound from the corrosion inhibitor formulation and investigate the synergistic effect of H_2S in the absence of the sulfur compound on C1018 carbon steel. H_2S is naturally occurring in the well and the ratio of partial pressure of CO_2 to partial pressure of H_2S has an impact on the corrosion rate [6], [14]. Hence the objective is to observe if H_2S functions as a sulfur source if it is present in small amounts. Two case scenarios were studied in order to investigate these many factors, including pH, temperature, and the effect of H_2S rate: case a) the presence of sulfur compound in a pure CO_2 environment and case b) the absence of sulfur compound in the CO_2/H_2S environment. The influence of each of these factors on the corrosion rate was determined using bubble tests and a rotating cage autoclave.

2 Literature Review

In the realm of oil and gas production, corrosion stands as a formidable adversary, posing significant challenges to infrastructure integrity, operational efficiency, and environmental safety [1]. This literature review delves into the multifaceted domain of corrosion control, encompassing various aspects ranging from the types of corrosion encountered in the industry to the corrosion evaluation methodologies. This chapter seeks to provide an overview of corrosion mechanisms, various types of corrosion inhibitors, and corrosion testing procedures. This review aims to shed light on current cutting-edge approaches and new trends in corrosion mitigation, providing useful insights for engineers, and practitioners in the field.

2.1 Introduction to corrosion

Corrosion is an electrochemical process that causes localized anodic and cathodic reactions on a metal's surface. Figure 2.1 illustrates the basic chemical corrosion processes [6]. When metal is exposed to a corrosive solution (electrolyte), the protons (from carbonic acid) are reduced at the cathode with the liberation of ferrous ions at the anode to maintain electroneutrality. The cathode and anode exchange positive and negative charges through the electrolyte. Positively charged ions are discharged into the electrolyte and can bind with negatively charged groups [15].

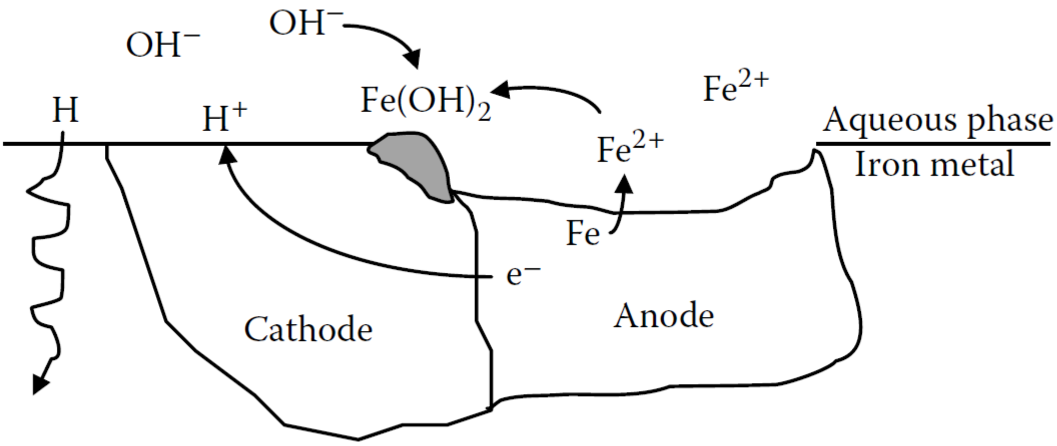


Figure 2.1 Reactions in corrosion cell [6]

Natural gas and crude oil can both contain a variety of highly impure, naturally corrosive compounds. Corrosive media commonly found in oil and gas wells and pipelines include CO_2, H_2S, and free water. When water reacts with CO_2 and H_2S, the following reactions occur: [15]

$$H_2CO_3 \text{ reaction: } Fe + H_2CO_3 \rightarrow FeCO_3 + H_2 \qquad (R\ 2.1)$$

$$H_2S \text{ reaction: } Fe + H_2S + H_2O \rightarrow FeS + 2H^+ \qquad (R\ 2.2)$$

These resulting molecules adhere to the cathode or release into the electrolyte, thereby causing the corrosion process to continue [15]. Corrosion can be classified based on the mechanism, type of the corrodent's appearance, and causes that encourage corrosion, resulting in diverse forms such as general corrosion, localized corrosion, stress corrosion cracking, and microbially influenced corrosion [16].

2.1.1 Sweet Corrosion (CO_2 corrosion)

In the oil and gas industry, the most prevalent type of corrosion is sweet (CO_2-induced). CO_2 gas is highly soluble in water, including saline water, and slightly more so in hydrocarbons. Hydrocarbon fluids are typically formed in contact with water, and reservoirs often include high levels of CO_2. CO_2 dissolves in the aqueous phase of the hydrocarbon product, leading to corrosion of carbon steel. When CO_2 gas dissolves in water, bicarbonate, carbonic acid, and hydrogen ions are produced [7]. The following are the main cathodic corrosion reactions that lead to the evolution of hydrogen [17]:

$$2H^+_{(aq)} + 2e^- \rightarrow H_{2(g)} \tag{R 2.3}$$

$$2H_2CO_{3(aq)} + 2e^- \rightarrow H_{2(g)} + 2HCO_3^-{}_{(aq)} \tag{R 2.4}$$

$$2HCO_3^-{}_{(aq)} + 2e- \rightarrow H_{2(g)} + 2CO_3^{2-}{}_{(aq)} \tag{R 2.5}$$

The metal dissolution at the anode proceeds in the following manner [17]:

$$Fe_{(s)} \rightarrow Fe^{2+}_{(aq)} + 2e^- \tag{R 2.6}$$

At low temperatures, sweet corrosion is commonly attributed to the formation of iron carbonate ($FeCO_3$) [18]. Corrosion is controlled by the rate of iron ion generation on surfaces covered in iron carbonate at temperatures below 60 °C and pH levels below 4 [7]. However, at temperatures above 150 °C, sweet corrosion of Fe-based alloys can produce Fe_3O_4 [18]. CO_2 localized corrosion can be classified into three types based on stagnant/flowing conditions and fluid velocity: pitting, mesa attack, and flow-induced corrosion [7].

2.1.2 Sour Corrosion (H_2S corrosion)

Sour corrosion is caused when the metal encounters hydrogen sulfide and moisture, causing the most harm to drill pipes. While H_2S does not have corrosive properties on its own, the addition of water causes it to become extremely corrosive, which can cause pipeline embrittlement. When dissolved in water, hydrogen sulfide is a weak acid that release hydrogen and bisulfide ions and can cause corrosion [7], [15]. The dissociation of H_2S is explained in the following reactions [19]:

$$H_2S_{(g)} \leftrightarrow H_2S_{(aq)} \qquad (R\ 2.7)$$

$$H_2S_{(aq)} \leftrightarrow H^+ + HS^- \qquad (R\ 2.8)$$

$$HS^- \leftrightarrow H^+ + S^{2-} \qquad (R\ 2.9)$$

Hydrogen and iron sulfides (FeS_X) are the byproducts of corrosion. At low temperatures, iron sulfide scale can act as a barrier against gradual corrosion. Sour corrosion can be present as uniform, pitting, or stepwise cracking [15].

2.2 Corrosion Inhibitors

Corrosion inhibitors are the most commonly used method to prevent interior corrosion in oil and gas pipelines [7]. They provide a protective coating that prevents metallic items from corroding when in contact with corrosive conditions. They are either organic or inorganic and are developed with the capability to adsorb onto the metal surface, creating a barrier film, or neutralizing the detrimental effects of corrosive substances. Film-forming corrosion inhibitors (FFCI) are used more frequently as they form impermeable hydrophobic layer that prevents damaging materials from accessing the metal surface, thus delaying the process of corrosion. In other words, the cathodic reduction of protons and subsequently the anodic liberation of ferrous ions limits access of the protons to the metallic surface hence mitigating the corrosion process[20], [21]. The following are some common film-forming inhibitors [7]:

- Amide
- Amide/Imidazoline
- Amide/Imidazoline + quarternary salts
- Amines
- Amine Salts
- Ethoxylates
- Phosphonate esters
- Quaternary ammonium salts
- Sulfonates

Film Forming Corrosion Inhibitors (FFCIs) are beneficial for the prevention of CO_2 and H_2S corrosion and their effectiveness is dependent on their ability to adhere to metal surfaces and form a protective coating [6], [7]. By acting as a barrier, this layer stops corrosive chemicals like water and chloride ions from penetrating the metal surface. FFCIs come in a variety of molecular forms; they can be tiny molecules or polymers. However, majority of are organic amphiphiles that have a hydrophobic tail and a polar headgroup. The hydrophobic tails of these compounds attract liquid hydrocarbons, generating an oily layer that improves protection against corrosive aqueous phases, while their polar headgroups interact with iron atoms on the surface. When the hydrophobic tail is sufficiently long, a protective double layer of surfactant FFCI can be formed. However, under certain multiphase flow conditions, localized corrosion may occur due to areas of the pipe wall remaining unprotected by FFCIs. For instance, corrosion on the upper segment of a pipeline resulting from condensed water mixed with CO_2/H_2S/organic acids poses a challenge, as the inhibitor may fail to adequately reach these vulnerable areas [6].

The corrosivity of fluids is dependent on various factors such as sour gases, temperature, pressure, bacteria, pH, solids, and fluid velocity. The structure of the corrosion inhibitor generally includes a long-chain hydrocarbon structure. There are different methods proposed for the optimization of these components and varying their physical properties [7].

2.2.1 Phosphate esters

Phosphate esters, including monoesters and diesters, serve as effective film-forming corrosion inhibitors (FFCIs) and are often combined with other types of FFCIs. They are synthesized by reacting alcohols, alkylphenols, or their alkoxylated derivatives with phosphating agents like phosphorus pentoxide or orthophosphoric acid. This reaction produces a mixture of monoesters and diesters, which exhibit different levels of hydrophilicity and partition between liquid hydrocarbon and water phases. Phosphate esters containing hydrophobic nonylphenol groups demonstrate superior effectiveness as FFCIs compared to linear or branched aliphatic phosphate esters [6]. Nonylphenol diesters show greater efficacy than their monoester counterparts. However, phosphate esters can form insoluble salts with Fe(II) and Ca(II), potentially causing deposition on pipe walls and impeding further corrosion. Phosphate esters derived from poly-oxyalkylated thiols, such as octyl or dodecyl mercaptan reacted with varying amounts of ethylene oxide, are particularly useful as pitting FFCIs, especially in deep gas wells. Poly(oxy-1,2-ethanediyl) tridecyl hydroxy phosphate is identified as a preferred FFCI in formulations with quaternary ammonium salt FFCIs and thiocarbonyl compounds. Additionally, amine salt reaction products of phosphate esters with various amines, including acylated polyamines, morpholine, and ethoxylated fatty amines, are claimed to inhibit general corrosion and cracking-type corrosion while offering improved environmental properties [6].

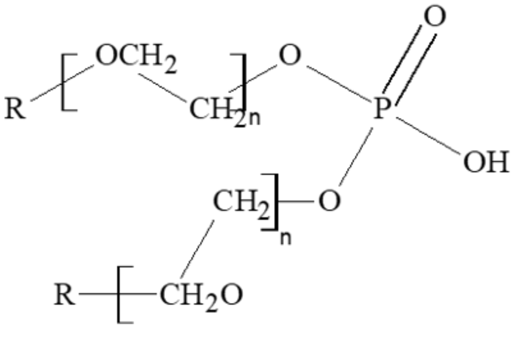


Figure 2.2 Typical structure of phosphate ester corrosion inhibitors.

2.2.2 Imidazolines

Imidazolines represent a prevalent and extensively researched class of corrosion inhibitors in the oil and gas sector, demonstrating efficacy even in challenging high-pressure, high-temperature conditions [6]. They are negatively charged cationic surfactants with imidazoline cores that do not vary with pH, making them easily adsorbed on negatively charged metal surfaces [7]. These compounds are synthesized by condensing a polyamine with a carboxylic acid, yielding various products including 2-alkylimidazolines and amido imidazolines. Imidazoline-based corrosion inhibitors can undergo protonation or alkylation to form hydrophilic imidazolinium salts with corrosion inhibition properties. Studies suggest that the hydrophobic chain length significantly impacts the inhibition efficiency of imidazolines. In numerous imidazoline-based Film Forming Corrosion Inhibitors (FFCIs), there are additional groups attached to one of the nitrogen atoms within the ring structure. These groups often incorporate nitrogen atoms, typically sourced from polyamines like diethylenetriamine or triethylenetetramine used in imidazoline synthesis. While these extra nitrogen atoms (or other heteroatoms with lone pairs of electrons) may potentially interact with metal surfaces, boosting FFCI adsorption, it's been found that having such pendant groups with heteroatoms (nitrogen, oxygen, or sulfur) and nonbonding electron pairs isn't a prerequisite for effective corrosion inhibition. Moreover, certain derivatives, like zwitterionic acrylated imidazolines, exhibit unexpectedly high inhibition performance. However, it's noted that imidazoline-based corrosion inhibitors can pose high acute toxicity, although efforts have been made to develop less toxic derivatives. Blends with quaternary ammonium salts are highlighted for their enhanced effectiveness [6].

Imidazoline is a highly effective film-forming combination used in numerous patents. In nature, they have limited solubility in water. Organic acids are used to neutralize corrosion inhibitors and boost their efficiency, resulting in cationic characteristics. Figure 2.3 illustrates the various imidazoline compounds utilized in corrosion inhibitor compositions [7].

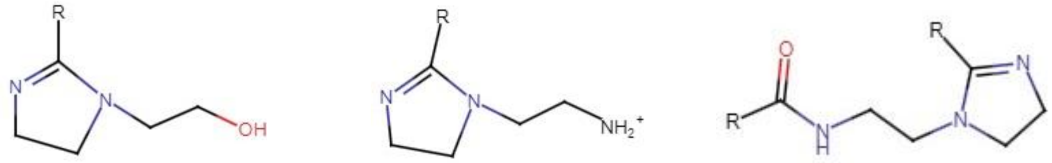


Figure 2.3 Different imidazoline compounds.

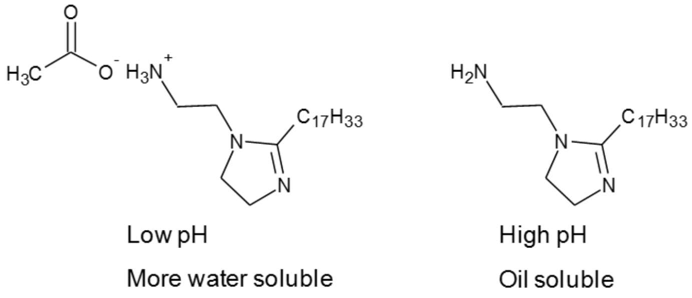


Figure 2.4 Imidazoline reacted with acetic acid (protonated) and unreacted.

2.2.3 Quaternary ammonium compounds

Quaternary ammonium salts (QASs), also referred as quaternary surfactant, is an organic corrosion inhibitor that forms a protective film to shield the metal surface from corrosive chemicals [7]. Although many of them are also effective as biocides, which can help prevent biofilm formation and hence under deposit corrosion, they are typically highly toxic. It is reported that didecyldimethyl ammonium chloride is the favored quaternary amines FFCI for downhole applications. The structure of didecyldimethyl ammonium chloride is shown in Figure 2.5. It can be combined with other ingredients such phosphate esters and thiocarbonyl compounds, or used alone [6]. In one of the studies, the synergistic effect of thiourea and imidazoline-based dissymmetric bis-quaternary ammonium salts for CO_2 corrosion control at elevated temperatures was investigated. The findings highlighted the necessity of ongoing research into corrosion prevention techniques, especially in tough situations such as sour environments, where the presence of H^+ ions can increase corrosion. Because of their cationic properties and capacity to establish strong interactions with metal surfaces, quaternary ammonium compounds present a promising means of mitigating corrosion in a variety of industrial environments [7].

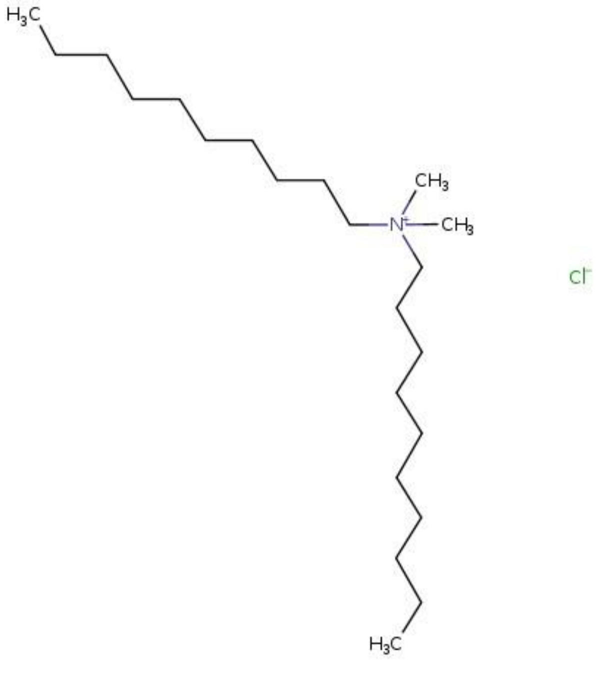


Figure 2.5 Structure of didecyldimethyl ammonium chloride [22].

2.2.4 Sulfur compounds

Various sulfur compounds, such as thiosulfate ions and mercaptocarboxylic acids, have proven effective as synergists for inhibiting corrosion, particularly in environments prone to cracking corrosion. The oxidation of mercapto groups to disulfide forms has been identified as the mechanism behind their corrosion inhibition. Notably, compounds like 3,3′-

dithiodipropionic acid (DTDPA) and mercaptoethanol (ME) demonstrate promising inhibition properties, with ME being especially effective against both general and localized corrosion. Patents highlight sulfur compounds as corrosion inhibitors, with trithiones being particularly noted for their effectiveness in CO_2-corrosive environments. Additionally, thiophosphorus compounds have shown promise in preventing naphthenic acid corrosion, especially at elevated temperatures downstream. Sulfur-nitrogen compounds, including benzothiazoles and ether carboxylic acids, have been explored for inhibiting corrosion, with some promising results on ferrous metals. Thiocarboxylic acids, although effective, face challenges due to unpleasant odors, leading to research into alternative volatile inhibitors like 2,5-dihydrothiazoles and thiazolidines. Non-surfactant compounds like imidazolidinethiones and polyfunctional polymers are emerging as low-toxicity, biodegradable options for corrosion inhibition. Additionally, salts of amidomethionine derivatives and amines offer increased biodegradability and reduced toxicity, presenting a promising avenue for future FFCI formulations [6].

2.2.5 Environmental concerns of sulfur compounds

The use of toxic compounds in corrosion inhibitors such as sulfur compounds, has raised environmental and health concerns. 2-Mercaptoethanol (CAS 60-24-2) is a chemical compound represented by the formula $HOCH_2CH_2SH$ and is derived from a combination of ethylene glycol ($HOCH_2CH_2OH$) and 1,2-ethanedithiol ($HSCH_2CH_2SH$). Its widespread use is due to its hydroxyl group, which enhances solubility in water and reduces volatility. The synthesis of 2-mercaptoethanol typically involves the reaction of hydrogen sulfide with ethylene oxide. 2-ME causes irritation to the nasal passageways and respiratory tract upon inhalation, leading to symptoms such as shortness of breath. The chemical compound has a notably low odor threshold (0.12–0.64 ppm) and prolonged exposure can induce hepatotoxicity and pose risks to aquatic organisms [12]. Thioglycolic acid, also known as mercaptoacetic acid is toxic corrosive. Ingestion of the chemical can irritate the mouth and throat, causing nausea, vomiting, and possibly diarrhea [23], [24]. Whereas, sodium thiosulfate is an inorganic chemical and functions as both a ligand and a reducing agent [25] Thiosulphate is also a commonly available sulfur source for bacteria and yeast development which can lead to microbial influenced corrosion [13], [26]. A study also discovered that high concentrations of sodium thiosulfate caused significant fish mortality and pathological changes in critical organs such as gills [27]. Hence, these sulfur compounds can affect personnel working in the warehouse who are handling corrosion inhibitors containing sulfur compounds, as well as marine species.

2.3 Corrosion testing and evaluation methods.

Leading corrosion inhibitor manufacturers face a significant obstacle in conducting laboratory testing that replicates real field conditions. Under a variety of operating situations, including fluid regimes, varying oil-to-water ratios, varied surface qualities, etc., a good corrosion inhibitor should, in general, function to prevent corrosion in pipelines. To assess the corrosion inhibitor for field applications, it is essential to conduct a set of laboratory tests that evaluate the performance of the inhibitor. The most common laboratory tests for selection of corrosion inhibitor include bubble, rotating cylinder electrode, rotating disc electrode, jet impingement, and rotating cage electrode [7]. The focus in this study is on the bubble test and rotating cage electrode, which will be further explored. In addition, electrodes and coupons are utilized for corrosion monitoring.

2.3.1 Corrosion testing procedures.

2.3.1.1 Bubble test.

Bubble/kettle testing evaluates corrosion inhibitor efficacy in the absence of flow. The test is appropriate for rapidly performing a large number of tests, such as the first stage of corrosion inhibitor selection or screening a wide range of field conditions. In this method, the corrosion rate is monitored by either weight loss measurements or electrochemical method known as linear polarization resistance [7]. Linear Polarization resistance (LPR) is used to gauge the corrosion resistance of steel electrode. It measures the polarization resistance (R_p) at the steel/electrolyte interface around the Open Circuit Potential (OCP), representing the slope of the polarization curve tangent at OCP, typically in ohms (Ω cm^2). To calculate R_p, the potential is varied within OCP ± 10 mV at a rate of 0.2 mV/s. Corrosion rates are then computed for each sample, adjusting the Stern-Geary constant (B) to match the weight loss corrosion rate by integrating LPR readings. The formula used for this conversion is as follows,

$$CR\ (mm/y) = (11.597 \times B) / (R_p \times A) \qquad \textit{Equation 2.1}$$

where A represents the total area of the sample in cm^2. The LPR technique can be implemented using either a 3-electrode setup (reference, counter, and working electrodes) or a 2-electrode setup (two identical working electrodes only).

2.3.1.2 Rotating cage autoclave

Internal flow in pipes produces wall shear stresses, which contribute to the flow-accelerated corrosion (FAC) mechanism. Typically, the flow regime is turbulent, which increases the mass transfer of corrosive species to the metal surface. The rotating cage autoclave (RCA) is one of the most promising methods for modeling pipeline corrosion in the lab [28]. Rotating cage autoclaves are commonly used to determine the effectiveness of inhibitors against sweet corrosion under high temperature, pressure, and flow conditions [18]. One of the advantages of RCA is the wide range of achievable and applicable shear stresses on specimens, which can vary from 20 Pa to 200 Pa [28]. The equation for shear stress is specified by ASTM G170 standard [29] as follows:

$$\tau_w = 0.0791 \times Re^{-0.3} \times \rho r^2 \times \omega^{2.3} \qquad \textit{Equation 2.2}$$

$$Re = \frac{2\rho\omega r^2}{\mu} \qquad \textit{Equation 2.3}$$

Where r is the cage radius (m), Re is the Reynolds number, ρ is the fluid density (kg/m^3), ω is the rotational speed (rad/s), and μ is the dynamic viscosity (kg/m/s). Rotating cage technology effectively evaluates both uniform and localized corrosion. Additionally, the technique has demonstrated a strong correlation in terms of reproducing the inhibitor's in-field performance [18].

2.3.2 Corrosion evaluation methods

2.3.2.1 Weight loss measurement

Weight loss is a corrosion evaluation technique that measures a material's corrosion rate by comparing pre-corroded specimens to a cleaned, corroded specimen. This data can be used to calculate inhibitor efficiency and corrosion rate. However, it's not reliable for identifying corrosion attack mechanisms or local attacks. To better understand these mechanisms and quantify local attacks, weight loss should be combined with other characterization techniques [18].

2.3.2.2 Scanning Electron microscopy

Understanding the compositions, crystal structures, and attack morphologies of corrosion products is essential to recognize the corrosion mechanisms. This information can be obtained from scanning electron microscopy (SEM). SEM images are generated by scanning a material with a concentrated electron beam. This is used to qualitatively examine the topography of the coupons (pitting corrosion), morphology, and composition [18].

2.3.2.3 Surface profilometry

Although research utilizing electrochemical methods are highly valuable in comprehending issues linked to corrosion, their ability to capture local corrosion attacks like pitting is limited. To identify local corrosion attacks such pitting, surface profilometry or a vertical scanning interferometer (VSI) can be used. For a corrosion attack to be classified as pit or local, the ratio of the deepest depth of attack to the average penetration must exceed five [18].

3 Material and methods

This chapter outlines the methodologies and materials utilized to acquire the experimental findings detailed in this report. All experiments were conducted within SLB's laboratories.

3.1 Materials

3.1.1 Brine composition

Synthetic brine was prepared by combining 30 g of analytical grade NaCl salts with 1000 mL of distilled water. Two sets of brine were made, and the pH of the solution was adjusted to 4 and 6 with $NaHCO_3$ and CO_2 partial pressure.

3.1.2 Additives

The filming agents, which were phosphate ester, imidazoline, and quaternary amines, were composed of 20% w/w of the active ingredient dissolved in an acetic acid and water solution. To prepare the sulfur compounds, namely thiosulfate (TS), thioglycolic acid (TGA), and 2-mercaptoethanol (ME), 5% w/w of the active ingredient was blended with water. The molecular structure of the sulfur compounds is shown in Figure 3.1 [30], [31], [32].

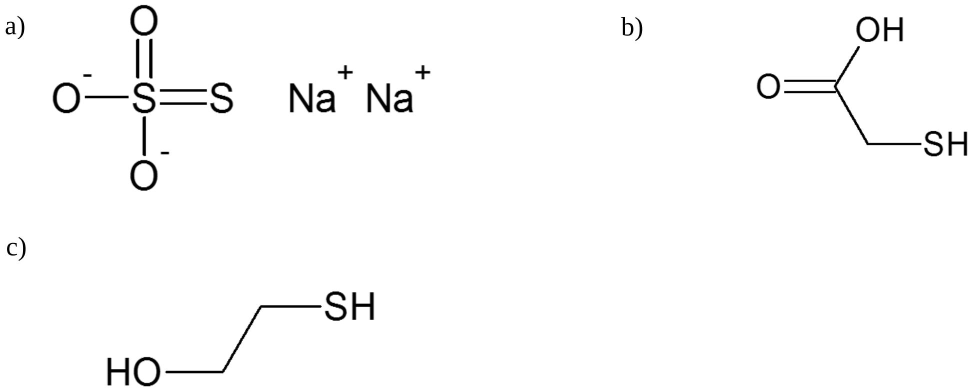


Figure 3.1 Molecular structure of sulfur compounds: a) Thiosulfate, b) Thioglycolic acid, c) 2-Mercaptoethanol.

3.1.3 Corrosion coupons

Carbon steel (C10180) electrodes and coupons were used during this experiment. The chemical composition is specified in Table 3.1 [33]. Coupons with dimensions of 25x15x1.5 mm, a density of 7.85 g/cm^3, and an area of 8.7 cm^2 were utilized. The electrodes used have an area of 2.365 cm^2 and a density of 7.87 g/cm^3.

Table 3.1 Chemical composition of C1018 [33].

Element	**Content (%)**
Manganese, Mn	0.60-0.90
Carbon, C	0.15-0.20
Sulfur, S	0.05 (max)
Phosphorous, P	0.04 (max)

3.1.4 Cleaning solution

To improve reproducibility and to reduce error or any contaminants, the equipment were cleaned before and after each experiment using dish soap and distilled water, and then dried with acetone. Prior to the experiment, the coupons were cleaned with acetone and then after the experiment with Clark solution. It is critical to be able to remove any corrosion product that forms on the surface of a metal coupon during testing. This is essential, for example, to precisely quantify the rate of metal loss, as well as the degree and shape of corrosion attack. The Clarke's solution used in the tests was produced according to ASTM G1 [34], with 1000 mL of hydrochloric acid (specific gravity 1.19), 20 g antimony trioxide (Sb_2O_3), and 50 g stannous chloride ($SnCl_2$) [35].

3.2 Experimental setup

3.2.1 Bubble/Kettle test

Bubble/kettle test is the most common method to evaluate the efficiency of chemical corrosion inhibitors in a bulk solution. This test aims to determine the influence of corrosion inhibitor on corrosion rate under static conditions using linear polarization resistance. The tests are carried out on a brine solution to test various test conditions and the performance is evaluated by comparing corrosion rates before and after addition of inhibitor.

Main instruments and equipment for corrosion experiment include a kettle with a capacity of 1000mL, LPR probes, sparging tubes, hot plate/magnetic stirrer with thermocouple, and potentiostat. The experimental setup of bubble test is shown in Figure 3.2. The test was performed on a brine consisting of 3% NaCl. The test cell was sparged with CO_2 for 1 hour prior to logging corrosion rate data to remove oxygen from the system and simulate pipeline conditions. The electrodes were attached to the LPR probe and then inserted into the kettle, thereby starting the data logging. The baseline corrosion rate was established over 2 hours, then the sulfur compound was injected. In experiments involving the use of H_2S gas, the gas was initiated at this specific time instead of introducing a sulfur compound. The rest of the procedures remain the same. After logging the corrosion rate for another 2 hours, filming agent was introduced into the cell. The corrosion rate was monitored over 22 hours, and the inhibitor performance was established by comparison of the inhibited corrosion rate to the baseline uninhibited corrosion rate.

The efficiency of the inhibitor was calculated as follows:

$$\% \, Inhibitor\ efficiency = \frac{(C_1 - C_2)}{C_1} \times 100 \qquad \textit{Equation 3.1}$$

where, C_1 is the uninhibited corrosion rate (blank/baseline corrosion rate, take the average corrosion rate between beginning data logging and treatment), and C_2 is the inhibited corrosion rate (corrosion rate 22 hours after treatment).

The test conditions are summarized in Table 3.2. The experimental test number and their test conditions are shown in Appendix Table B.1.

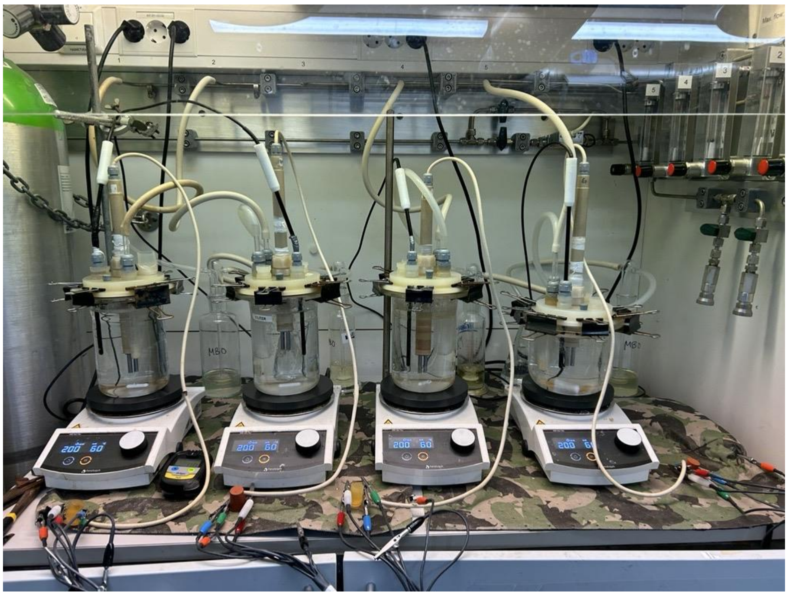


Figure 3.2 Experimental setup of bubble tests.

Figure 3.3 Gamry Instruments Potentiostat.

Table 3.2 Test Conditions for the bubble test

Brine	3% NaCl
Kettle volume, litre	1
Gas	100% CO_2
	99.99% /0.01% CO_2/H_2S
pH, adjusted with $NaHCO_3$ and CO_2 partial pressure	4 and 6
Temperature, °C	20 and 60
Duration, hours	22
Metal type	C1018
Filming agent, ppm as active	5
Sulfur compound, ppm as active	2

3.2.2 Rotating cage autoclave test

Rotating cage autoclave (RCA) is used to study the effect of corrosion inhibitor on corrosion rate under high-shear conditions using weight loss measurement or coupons. The test was conducted in an autoclave with a rotating cage containing a metal coupon under a carbon dioxide atmosphere for 72 hours. By adding high purity CO_2 and using internal heaters, the cell replicates pipeline temperature and CO_2 partial pressure conditions. The performance is evaluated by comparing the corrosion rates in tests conducted with and without a corrosion inhibitor.

Figure 3.4 Experimental setup of RCA test.

The experiment is divided into 3 stages: pre-conditioning, liquid transferring and setting the temperature and pressure. The schematic of the RCA methodology is described in Figure 3.4. In the pre-conditioning stage, the produced water sample (or synthetic brine) is preconditioned by continuous sparging with a mixture of 100 mole% CO_2 at 20 °C for 2 hours in a sealed glass vessel. The pH of the brine is measured and is adjusted to 4 due to CO_2 sparging. During this two-hour time, the reactor assembly is prepared for fluid transfer by mounting the metal coupons on the rotating cage and dosing corrosion inhibitor if necessary. After mounting the coupons and adding the inhibitor, the locking cuffs are attached, and the compression bolts are tightened to close the reactor. The final step before transferring the test liquid, the reactor is sparged with CO_2 for 10 minutes. Once the reactor has been built and sparged, 750 mL of the preconditioned generated water sample (or synthetic brine) is delivered into the reactor via the liquid input from the glass jar under pressure from the same gas composition as during the brine pre-conditioning stage. The reactor is then heated to the test temperature and the rotation of the coupons is set to the desired rotation speed. At this stage, the H_2S gas is introduced into the system as per the required test conditions. The continuous gas flow is maintained for the entire 72 hours. At the end of the test, the coupons are retrieved for inspection and weight loss measurement. The test matrix for the performed RCA tests is mentioned in Appendix Table B.2.

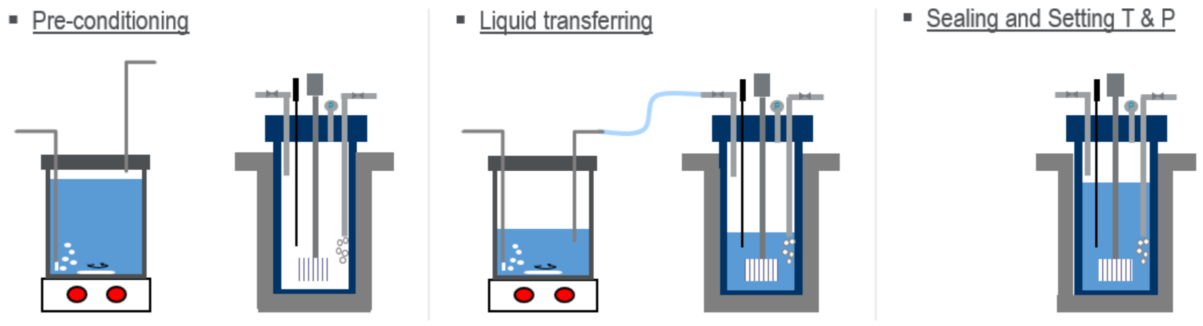


Figure 3.5 Schematic of the RCA methodology.

The test conditions for the RCA are summarized in Table 3.2.

Table 3.3 Test Conditions for the RCA test

Brine	3% NaCl
Kettle volume, ml	750
Gas	100% CO_2
	99.99% /0.01% CO_2/H_2S
pH, adjusted with CO_2 partial pressure	4
Temperature, °C	60

Rotation rate (rpm)	200
Duration, hours	72
Metal type	C1018
Filming agent, ppm as active	5
Sulfur compound, ppm as active	2

3.2.3 Corrosion evaluation method.

At the end of each experiment, the coupons are first cleaned with dish soap, followed by dipping them in Clark solution for 1 min and then with dish soap, distilled water and acetone. Weight loss measurements were conducted for each coupon and image analysis using optical microscope, profilometry and SEM was conducted for the required coupons.

3.3 Safety Procedures

The test methods and procedures for performing bubble test and rotating cage autoclave has been prepared in accordance with the precautions mentioned in Risk Assessment RA-063 and RA-064 respectively. The following procedures are followed:

- MSDS for all chemicals for the test method must be consulted.
- The H_2S safety course is a requirement for doing experiments utilizing the gas.
- Make sure that all leads and pipework are clean and do not come into contact with hot surfaces. Damaged leads can result in inaccurate measurements.
- Check the supply of carbon dioxide and hydrogen sulfide gas before screening to prevent the potential of the gas running out during testing.
- Ensure that the testing is conducted in a fume hood and well-ventilated space.
- Many corrosion inhibitors are hazardous to one's health, and even when used in small doses, adequate PPE should always be worn when working with them. The standard PPE include lab coat, safety goggles, gloves, breathing mask (while performing H_2S experiment) , and safety shoes.
- When conducting experiments using H_2S gas, it is necessary for the individual performing the tests to be accompanied by someone who has completed the H_2S safety training.

3.4 Simulation Software

3.4.1 ScaleSim

ScaleSim is an advanced thermodynamic simulation tool that integrates leading models to forecast various phase properties, including salt solubility, scale potential, pH levels, and gas solubility within oil/gas/water systems. By employing the Pitzer model alongside thermodynamic principles, ScaleSim accurately determines properties of the aqueous phase and the solubility of salts/minerals. Additionally, it utilizes a comprehensive Equation of State based PVT model to compute oil/gas phase properties and composition. Through seamless integration, ScaleSim facilitates the calculation of 3-phase equilibrium (water-oil-gas) and salt precipitation [36].

ScaleSim was used to simulate laboratory settings to forecast the pH and moles of CO_2 and H_2S in the aqueous phase. It was also used to model field pipeline conditions and convert them to laboratory test conditions.

3.4.2 NORSOK M-506

Norsok M506 is a corrosion model used in the oil and gas sector that is used for calculating corrosion rates in hydrocarbon production and process systems where the corrosive agent is CO_2. The model fits a large portion of the same laboratory data as the De Waard-Milliams model plus more experimental data at 100–150 °C. The model is freely accessible via the Norsok standards website and has been published as a standard for the Norwegian oil sector. The NORSOK model was used to predict the corrosion rate using the pressure, temperature, ionic strength and shear stress. The model was also used to calculate the shear stress with the input data such as flow rate, pipe diameter, pressure, temperature, and water cut. Although, there are limitations, the model does not consider H_2S in the calculation and hence is not suitable to predict sour corrosion [37].

4 Results and Discussion

The primary goal of the study was to determine whether a sulfur compound (SC) is necessary in field applications as much as it is in laboratory conditions to achieve effective corrosion inhibition. The experiments investigated the performance of filming agents based on phosphate esters, imidazoline and quaternary amines with and without SC in the absence and presence of H_2S. The results from various corrosion inhibitor monitoring and inspection methods are summarized in this chapter.

4.1 Corrosion monitoring with Bubble test

Among the several procedures used to examine corrosion prevention performance, the bubble test is particularly popular due to its simplicity and applicability. This section provides results from the bubble test experiments for various corrosion inhibitors. An example of the general trend obtained through the results is shown in Figure 4.1. The initial 2 hours of CO_2 sparging yields the baseline corrosion rate. After 2 hours, either the sulfur compound or H_2S is introduced into the system, resulting in a considerable decline in corrosion rate. After another 2 hours, filming agent is injected, and inhibition from both the sulfur compound and the filming agent is measured.

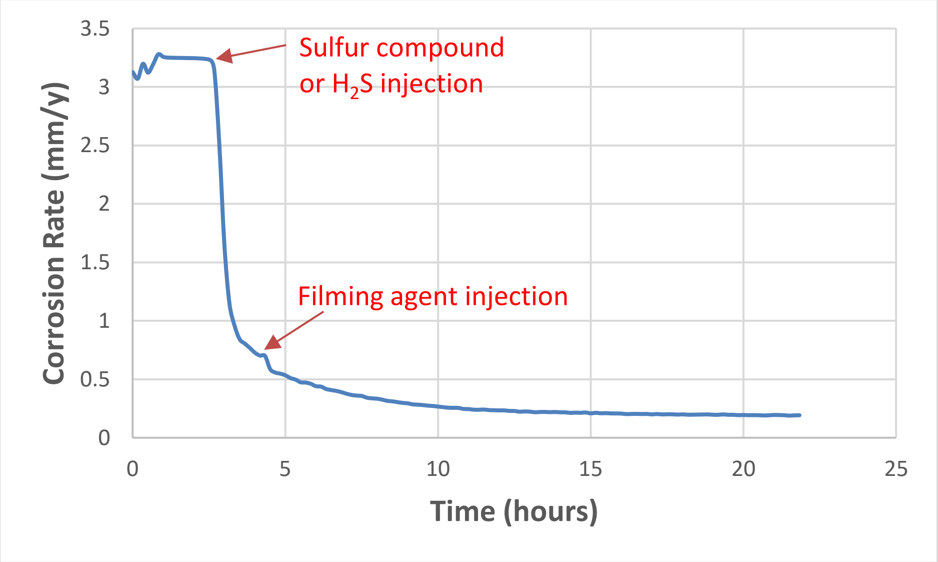


Figure 4.1 General trend of the corrosion rate trend obtained through the results.

4.1.1 Phosphate esters

Figure 4.2 represents the corrosion rate for phosphate esters with various sulfur compound and different test conditions. It was observed that phosphate ester molecule adsorbs better in the presence of sulfur compound in a CO_2 environment. In particular, presence of thioglycolic acid assisted the phosphate ester inhibitor by increasing the inhibition efficiency by 18.9% when compared to the performance of phosphate ester without any SC.

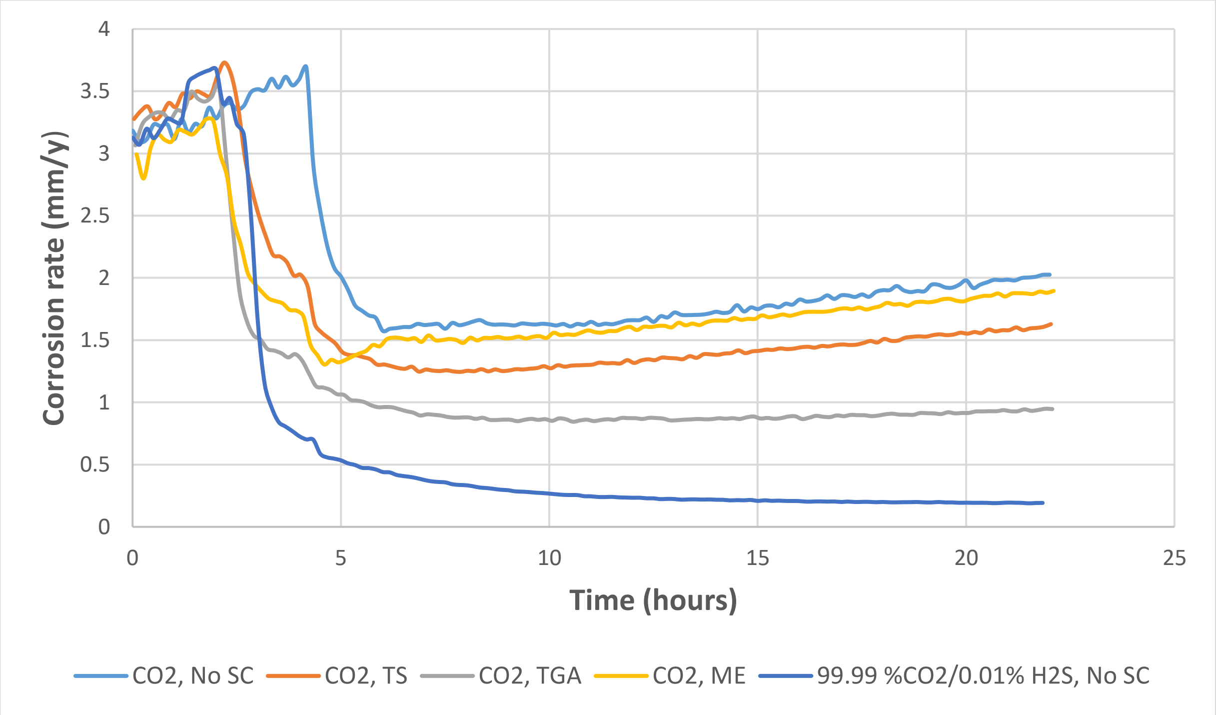


Figure 4.2 Performance of phosphate ester with and without sulfur compound at pH 4 and 60 °C.

However, in the presence of 0.01% H_2S gas, phosphate ester has shown stronger bond with the metal even in the absence of a sulfur compound providing better corrosion inhibitor efficiency of 94.9%.

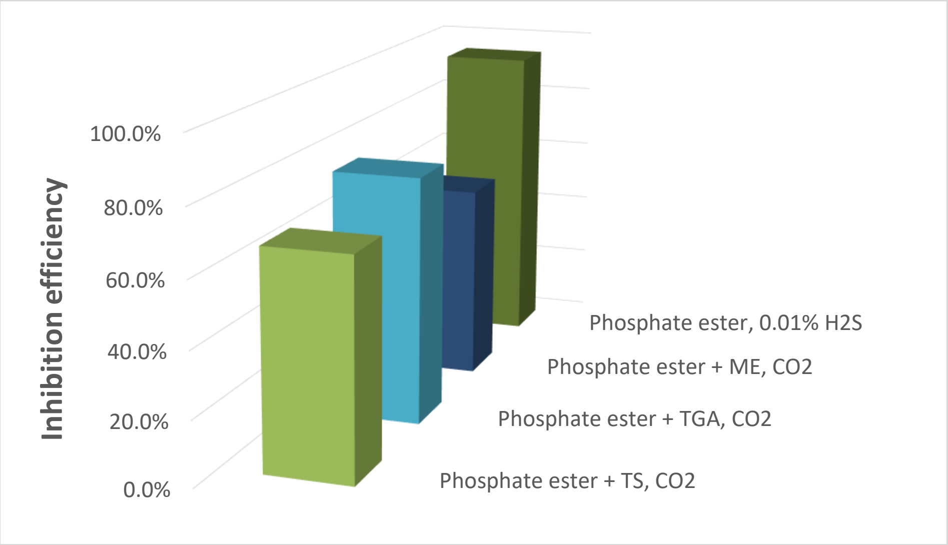


Figure 4.3 Inhibition efficiency of phosphate ester with and without sulfur compound at pH 4 and 60 °C.

4.1.2 Imidazoline

Corrosion rate for imidazoline in the presence of TS, TGA and ME obtained in the bubble test at 60 °C and pH 4 are shown in Figure 4.4. Comparing formulations with three different S-containing compounds reveals that product performance is heavily influenced by the type of the inhibitor formulations. The hydrolysis of imidazoline causes a rise in corrosion rates in environments that are CO_2 exclusive. The introduction of imidazoline, on its own, without the presence of any sulfur compounds, resulted in a marginal increase in the rate of corrosion. In a study in the presence of sulfur compounds, imidazoline's adsorption to the metal surface increased. TGA is a chelant and a strong reducing agent, that dissolves Fe_3O_4 and to a lesser extent FeO/$FeCO_3$. TGA and its iron complexes can oxidize to less soluble disulfides, resulting in a protective surface oxide/carbonate/disulfide coating. Iron disulfide layer adsorption occurs mostly at anodic locations (strong iron chelant) where iron is oxidized to $Fe^{2+/3+}$ (anodic inhibitor). Because of its rapid adsorption/absorption onto mild steel surfaces, it provides good inhibition even at low concentrations. Mercaptoethanol, despite lacking a carboxylate group, exhibits similar redox processes as TGA, resulting in a weaker ME-iron complex and a longer-lasting disulfide layer [9]. Nevertheless, imidazoline has demonstrated a stronger bond with the metal in the presence of 0.01% H_2S gas, even in the lack of a sulfur compound, offering superior corrosion inhibitor efficacy.

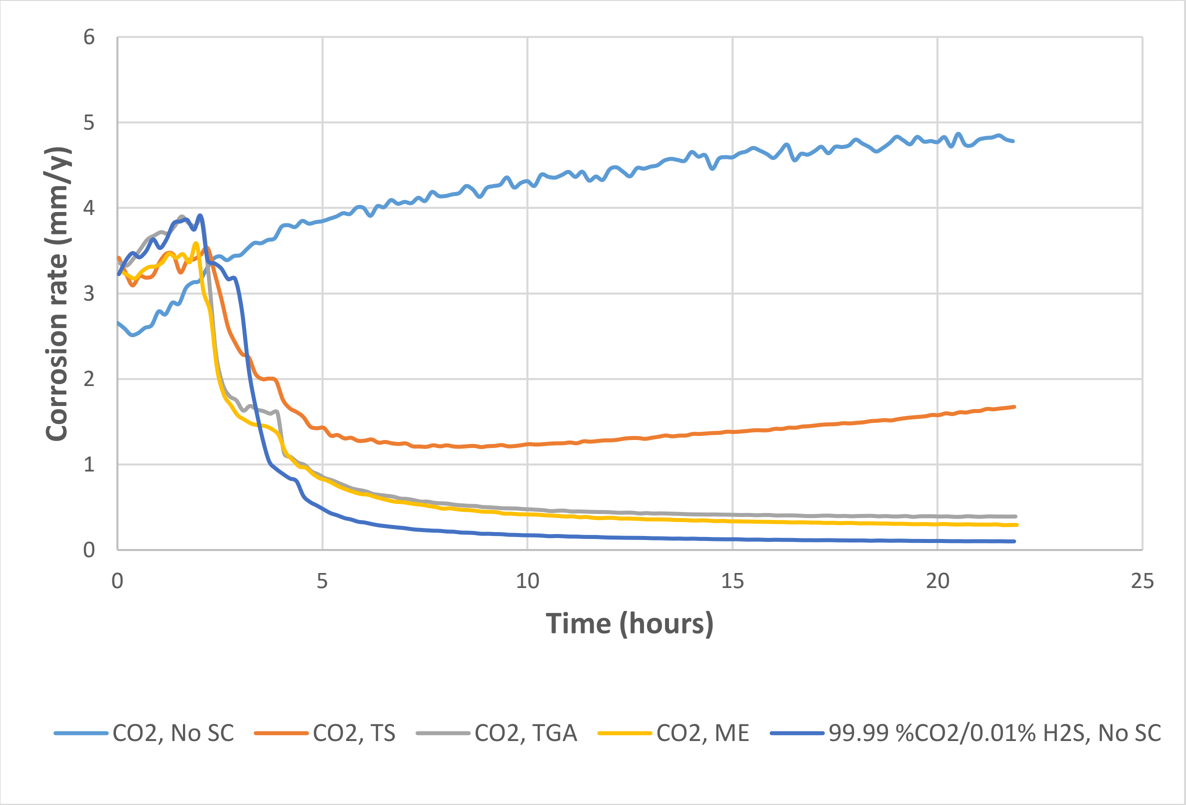


Figure 4.4 Performance of imidazoline with and without sulfur compound at pH 4 and 60 °C.

Figure 4.5 depicts the calculated inhibition efficiency based on the corrosion rate. According to the findings, imidazoline displayed an inhibitory efficiency of 97.4% in the presence of H_2S, which is much higher than imidazoline's efficacy in the absence of a sulfur source, which is 48.3%.

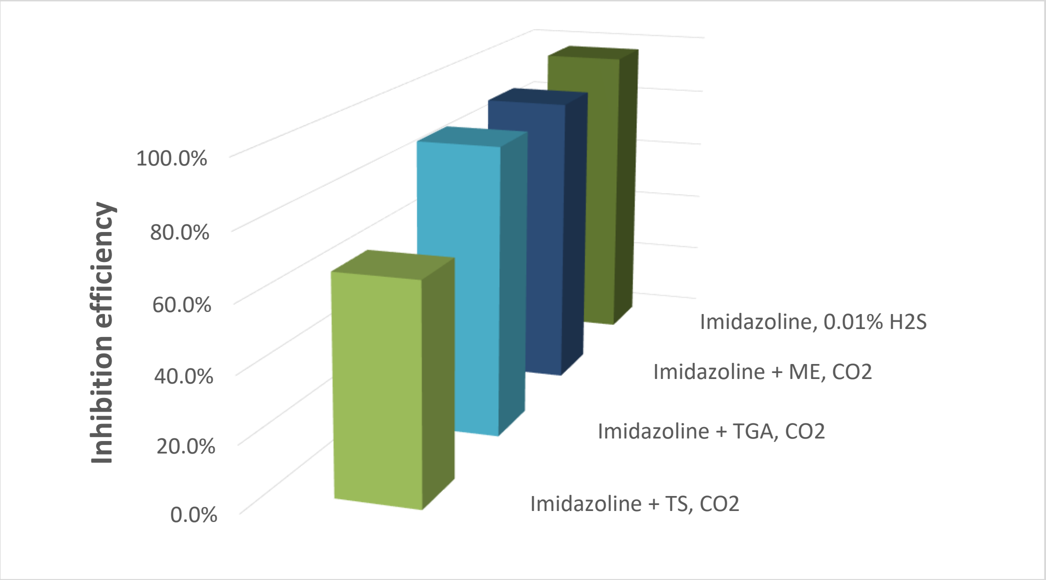


Figure 4.5 Inhibition efficiency of imidazoline with and without sulfur compound at pH 4 and 60 °C.

4.1.3 Quaternary amines

The bubble test results from the experiment with and without sulfur component in quaternary amines are displayed in Figures 4.6 and 4.7. It was noted that the rate of corrosion increased when the filming agent was injected without any sulfur ingredient. This demonstrated that in the absence of any sulfur compound, the anionic corrosion inhibitor did not adhere to the metal surface. The performance of quaternary amine in the presence of thiosulfate, thioglycolic acid, and 2-mercaptoethanol obtained the inhibition efficiency of 68.4%, 74.7%, and 81.4% respectively. Quaternary amines, on the other hand, adhered to the metal surface more readily and offered 86.4% corrosion inhibition when H_2S was present.

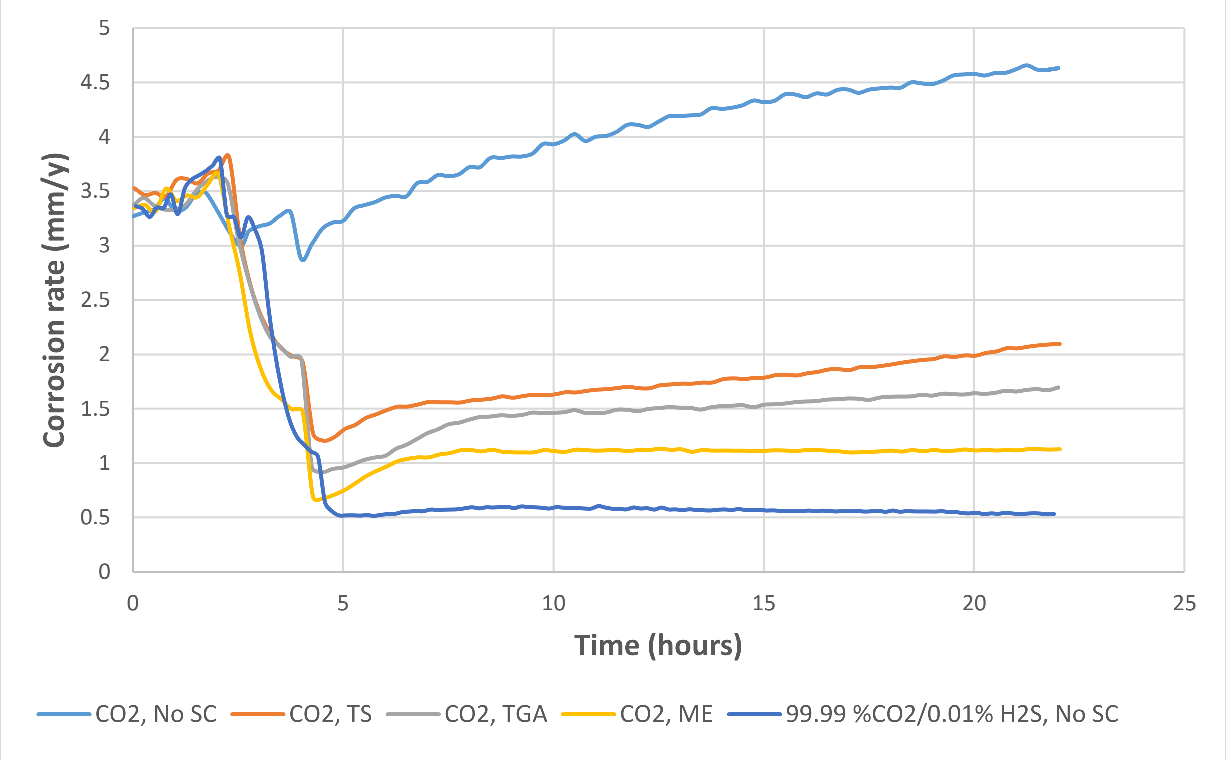


Figure 4.6 Performance of quaternary amines with and without sulfur compound at pH 4 and 60 °C.

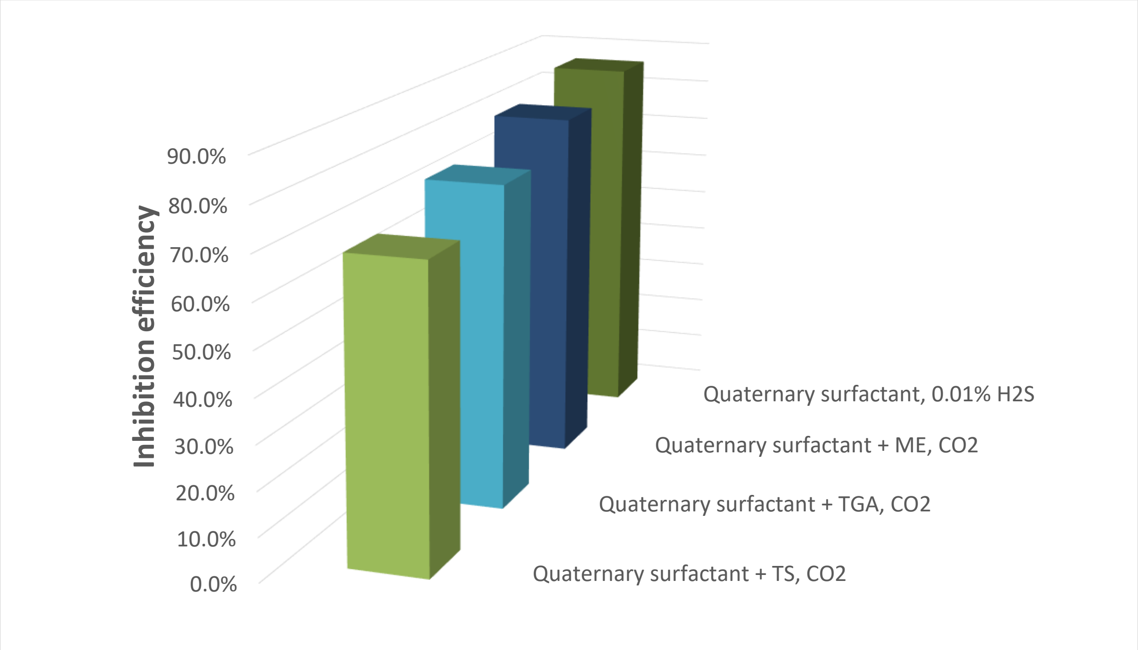


Figure 4.7 Inhibition efficiency of quaternary amines with and without sulfur compound at pH 4 and 60 °C.

4.1.4 Effect of pH

The pH of a solution has a considerable impact on the efficiency of corrosion inhibitors. Figure 4.8 demonstrates the corrosion inhibition efficiency of phosphate esters, imidazolines, and quaternary amines in the presence of 0.01% H_2S at pH 4 and pH 6. It was observed that the corrosion protection was better at higher pH (pH 6). The pH can alter the adsorption of corrosion inhibitors onto metal surfaces. The surface charge of the metal and inhibitor molecules varies with pH, influencing the adsorption process. Optimal pH circumstances may exist in which inhibitor molecules are more efficiently adsorbed onto the metal surface, resulting in a more stable protective layer. It is proposed that the resulting sulfur-iron complex is more stable at higher pH and follows the same trend as iron sulfide.

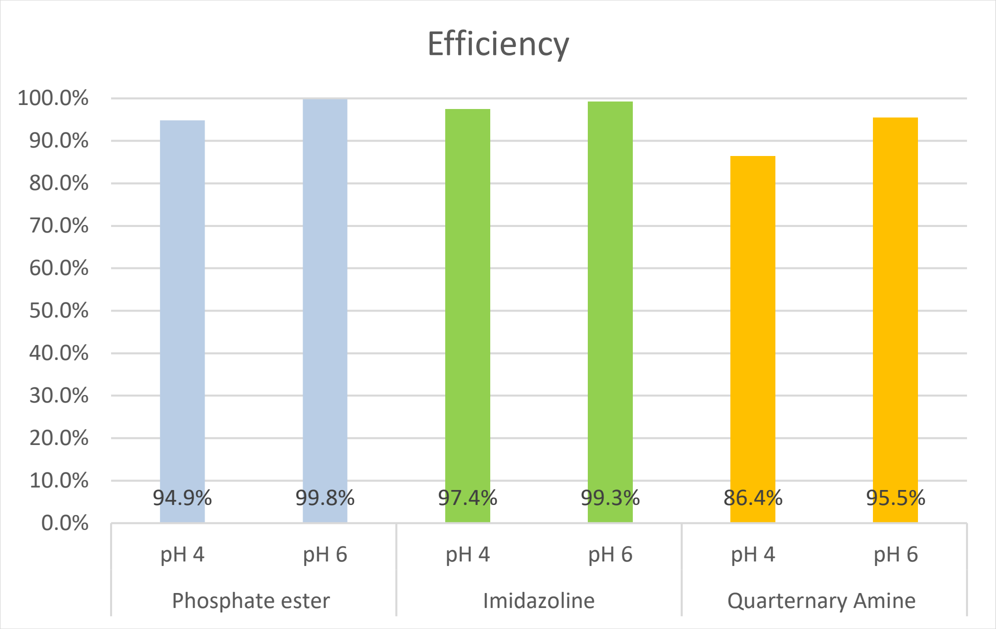


Figure 4.8 Performance of corrosion inhibitor without sulfur compound, in the presence of 99.99%CO_2/0.01%H_2S at 60 °C.

4.1.5 Effect of temperature

The temperature of a system is one of the factors that can significantly influence the effectiveness of corrosion inhibitors. In this study, two case scenarios were considered: case a) performance of corrosion inhibitor without sulfur compound in the presence of 99,99%CO_2/0.01%H_2S at pH 4, and case b) performance of corrosion inhibitor without sulfur compound in the presence of 99,99%CO_2/0.01%H_2S at pH 6, at 20 °C and 60 °C. The results obtained are shown in Figure 4.9 and Figure 4.10. It shows that elevated temperature can significantly restrict adsorption of quaternary amines, although will marginally restrict the adsorption for phosphate esters and imidazoline at pH 4. However, at pH 6, the effect of temperature is not quite significant. Because of the enhanced chemical reactions and higher mobility of reactant species, corrosion rates often rise with temperature. Higher temperatures can either accelerate or slow the adsorption process, depending on the inhibitor and the system. At high temperatures, the inhibitors may also experience thermal breakdown or deterioration, which would result in the loss of their inhibitive qualities.

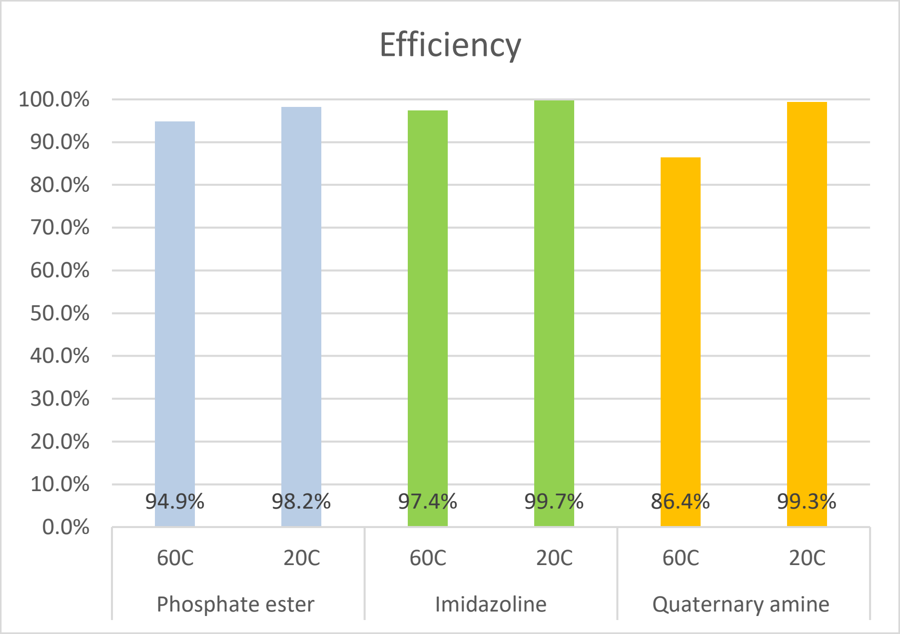


Figure 4.9 Performance of corrosion inhibitor without sulfur compound, in the presence of 99.99%CO_2/0.01%H_2S at pH 4.

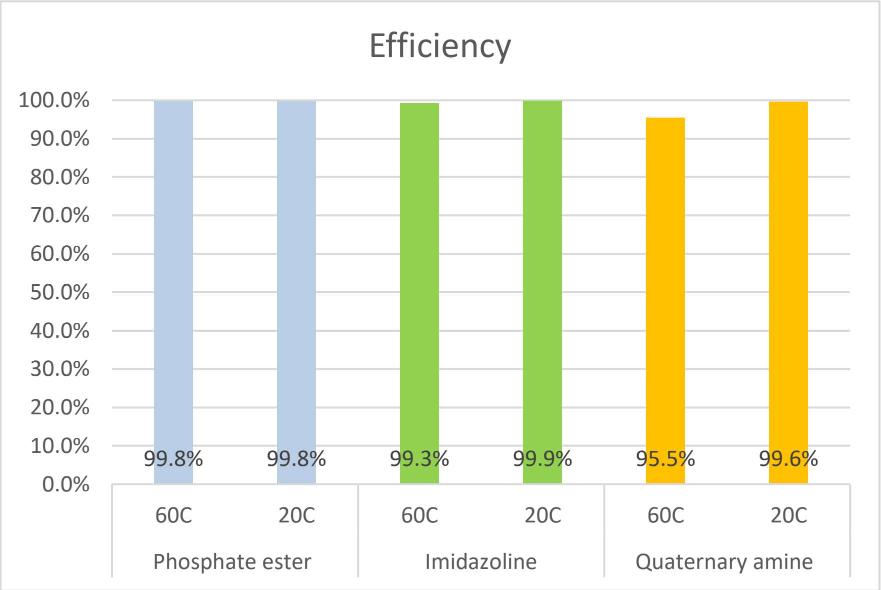


Figure 4.10 Performance of corrosion inhibitor without sulfur compound, in the presence of 99.99%CO_2/0.01%H_2S at pH 6.

4.1.6 Effect of H_2S rate

To understand the synergy effect of H_2S rate on the performance of corrosion inhibitors, the bubble test was performed in the presence of 0.005% H_2S and 0.01% H_2S and the results is shown in Figure 4.11. It was discovered that imidazolines performed similarly in the presence of 0.01% and 0.005% H_2S, with inhibitory efficacy of 97% and 92%, respectively. Similar results were obtained with quaternary amines when two distinct H_2S concentrations were present. Phosphate esters, on the other hand, performed better with 0.01% H_2S than with 0.005% H_2S. The synergistic impact of hydrogen sulfide (H_2S) on corrosion inhibitors is the phenomenon in which the presence of H_2S improves the efficacy of corrosion inhibitors. H_2S can form complexes with specific corrosion inhibitor molecules, modifying their chemical characteristics and increasing their propensity to adsorb on the metal surface. These complexes may have a stronger affinity for the metal surface or show more inhibitory efficacy than the inhibitor alone. In some situations, the presence of H_2S can aid in the creation of protective passive layers on metal surfaces, either by direct chemical reactions or by encouraging the adsorption of passivates [14]. Corrosion inhibitors may then interact synergistically with these passive layers, increasing their protective characteristics.

One of the key elements to consider while investigating the impact of H_2S on corrosion is the total amount of CO_2 and H_2S in the aqueous phase. This was ascertained by simulating the test conditions for various H_2S concentrations using the ScaleSim software. The outcomes are displayed in Figures 4.12 and 4.13. The total H_2S in the aqueous phase was 0.0037 mmol/kgH_2O when 0.01% H_2S was present, and 0.0018 mmol/kgH_2O when 0.005% H_2S was present. However, under both test conditions, the pH and total CO_2 in the aqueous phase do not vary, suggesting that those variables do not directly influence the test's outcomes.

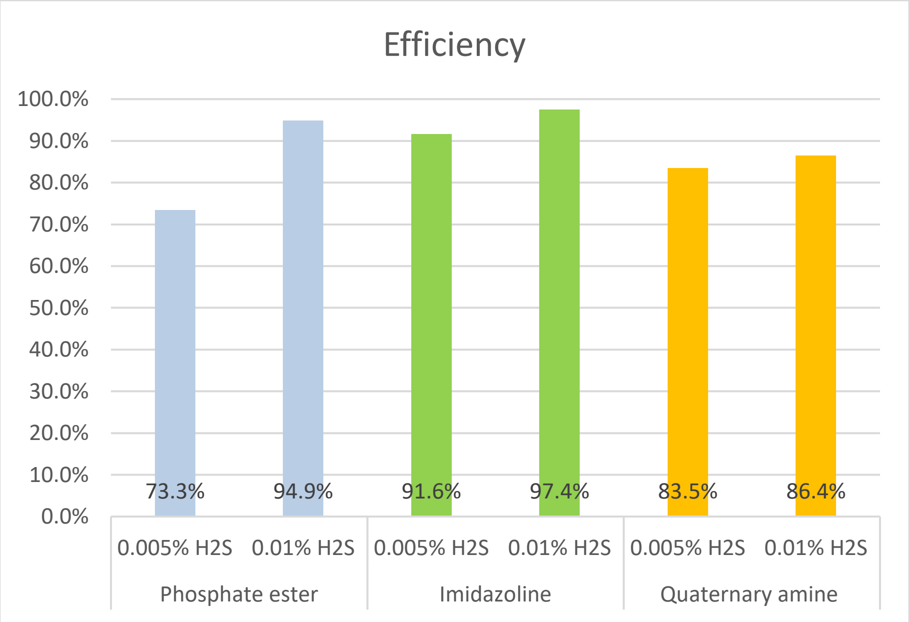
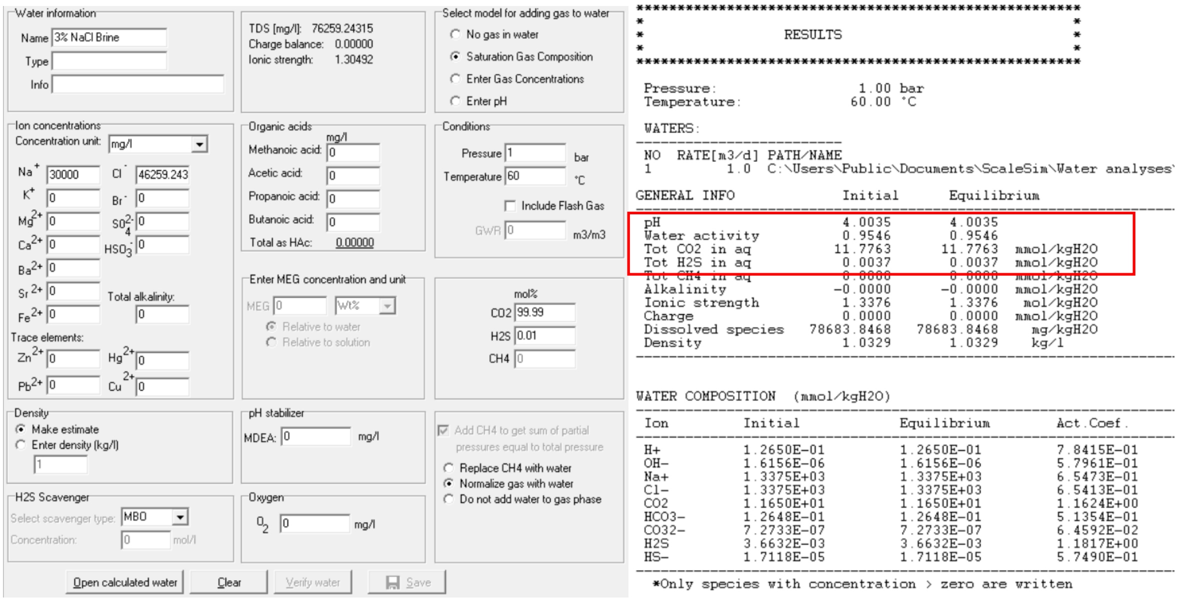


Figure 4.11 Performance of corrosion inhibitor without sulfur compound, in the presence of 99.99%CO_2/0.01%H_2S at pH 4 and 60 °C.



Name: 3% NaCl Brine
Type:
Info:

TDS [mg/l]: 76259.24315
Charge balance: 0.00000
Ionic strength: 1.30492

Select model for adding gas to water:
○ No gas in water
● Saturation Gas Composition
○ Enter Gas Concentrations
○ Enter pH

Concentration unit: mg/l
Na^+: 30000
Cl^-: 46259.243
K^+: 0
Br^-: 0
Mg^{2+}: 0
SO_4^{2-}: 0
Ca^{2+}: 0
HSO_3^-: 0
Ba^{2+}: 0
Sr^{2+}: 0
Total alkalinity: 0
Fe^{2+}: 0
Zn^{2+}: 0
Hg^{2+}: 0
Pb^{2+}: 0
Cu^{2+}: 0

Organic acids (mg/l):
Methanoic acid: 0
Acetic acid: 0
Propanoic acid: 0
Butanoic acid: 0
Total as HAc: 0.00000

Pressure: 1 bar
Temperature: 60 °C
☐ Include Flash Gas
GWR: 0 m3/m3

Enter MEG concentration and unit
MEG: 0 Wt%
● Relative to water
○ Relative to solution

mol%
CO2: 99.99
H2S: 0.01
CH4: 0

● Make estimate
○ Enter density (kg/l): 1

MDEA: 0 mg/l

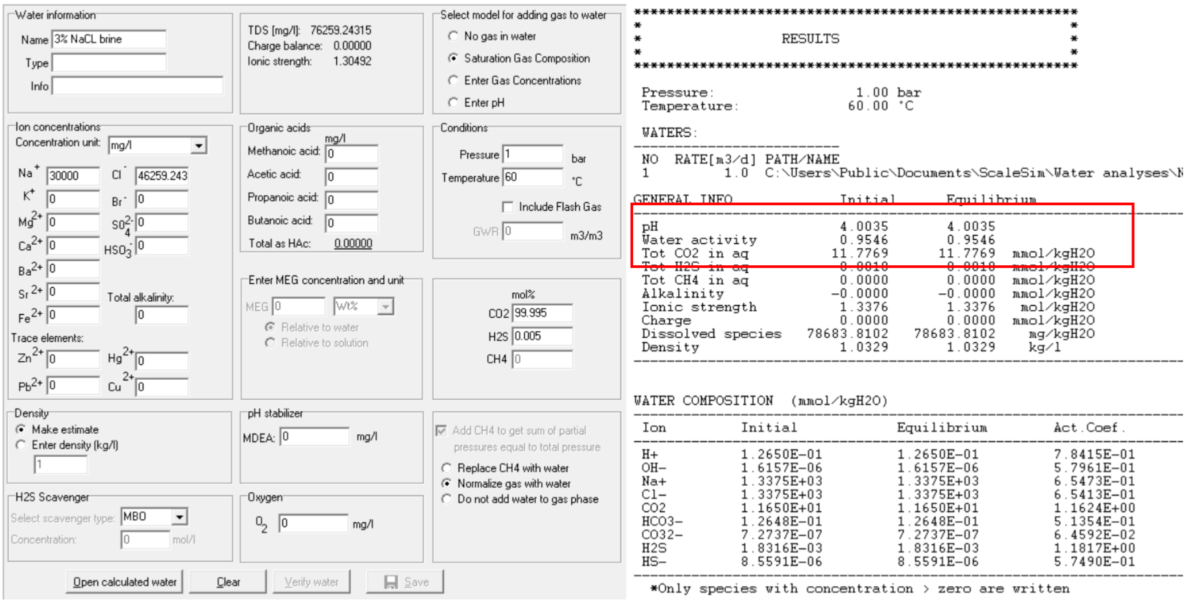
☑ Add CH4 to get sum of partial pressures equal to total pressure
○ Replace CH4 with water
● Normalize gas with water
○ Do not add water to gas phase

Select scavenger type: MBO
Concentration: 0 mol/l

O_2: 0 mg/l

```
*****************************************************
*                                                   *
*                  RESULTS                          *
*                                                   *
*****************************************************

 Pressure:                  1.00 bar
 Temperature:              60.00 'C

 WATERS:
-------------------------
 NO  RATE[m3/d] PATH/NAME
 1         1.0  C:\Users\Public\Documents\ScaleSim\Water analyses'

GENERAL INFO              Initial      Equilibrium
-------------------------------------------------------------------
 pH                        4.0035       4.0035
 Water activity            0.9546       0.9546
 Tot CO2 in aq            11.7763      11.7763  mmol/kgH2O
 Tot H2S in aq             0.0037       0.0037  mmol/kgH2O
 Tot CH4 in aq             0.0000       0.0000  mmol/kgH2O
 Alkalinity               -0.0000      -0.0000  mmol/kgH2O
 Ionic strength            1.3376       1.3376   mol/kgH2O
 Charge                    0.0000       0.0000  mmol/kgH2O
 Dissolved species     78683.8468   78683.8468    mg/kgH2O
 Density                   1.0329       1.0329    kg/l
-------------------------------------------------------------------

WATER COMPOSITION  (mmol/kgH2O)
-------------------------------------------------------------------
 Ion          Initial              Equilibrium          Act.Coef.
-------------------------------------------------------------------
 H+           1.2650E-01           1.2650E-01           7.8415E-01
 OH-          1.6156E-06           1.6156E-06           5.7961E-01
 Na+          1.3375E+03           1.3375E+03           6.5473E-01
 Cl-          1.3375E+03           1.3375E+03           6.5413E-01
 CO2          1.1650E+01           1.1650E+01           1.1624E+00
 HCO3-        1.2648E-01           1.2648E-01           5.1354E-01
 CO32-        7.2733E-07           7.2733E-07           6.4592E-02
 H2S          3.6632E-03           3.6632E-03           1.1817E+00
 HS-          1.7118E-05           1.7118E-05           5.7490E-01
-------------------------------------------------------------------
  *Only species with concentration > zero are written
```

Figure 4.12 Simulation input data and results for NaCl brine in the presence of 99.99%CO_2/0.01%H_2S.



Name: 3% NaCL brine
Type:
Info:

TDS [mg/l]: 76259.24315
Charge balance: 0.00000
Ionic strength: 1.30492

Select model for adding gas to water:
○ No gas in water
● Saturation Gas Composition
○ Enter Gas Concentrations
○ Enter pH

Concentration unit: mg/l
Na^+: 30000
Cl^-: 46259.243
K^+: 0
Br^-: 0
Mg^{2+}: 0
SO_4^{2-}: 0
Ca^{2+}: 0
HSO_3^-: 0
Ba^{2+}: 0
Sr^{2+}: 0
Total alkalinity: 0
Fe^{2+}: 0
Zn^{2+}: 0
Hg^{2+}: 0
Pb^{2+}: 0
Cu^{2+}: 0

Organic acids (mg/l):
Methanoic acid: 0
Acetic acid: 0
Propanoic acid: 0
Butanoic acid: 0
Total as HAc: 0.00000

Pressure: 1 bar
Temperature: 60 °C
☐ Include Flash Gas
GWR: 0 m3/m3

Enter MEG concentration and unit
MEG: 0 Wt%
● Relative to water
○ Relative to solution

mol%
CO2: 99.995
H2S: 0.005
CH4: 0

● Make estimate
○ Enter density (kg/l): 1

MDEA: 0 mg/l

☑ Add CH4 to get sum of partial pressures equal to total pressure
○ Replace CH4 with water
● Normalize gas with water
○ Do not add water to gas phase

Select scavenger type: MBO
Concentration: 0 mol/l

O_2: 0 mg/l

```
*****************************************************
*                                                   *
*                  RESULTS                          *
*                                                   *
*****************************************************

 Pressure:                  1.00 bar
 Temperature:              60.00 'C

 WATERS:
-------------------------
 NO  RATE[m3/d] PATH/NAME
 1         1.0  C:\Users\Public\Documents\ScaleSim\Water analyses\N

GENERAL INFO              Initial      Equilibrium
-------------------------------------------------------------------
 pH                        4.0035       4.0035
 Water activity            0.9546       0.9546
 Tot CO2 in aq            11.7769      11.7769  mmol/kgH2O
 Tot H2S in aq             0.0010       0.0010  mmol/kgH2O
 Tot CH4 in aq             0.0000       0.0000  mmol/kgH2O
 Alkalinity               -0.0000      -0.0000  mmol/kgH2O
 Ionic strength            1.3376       1.3376   mol/kgH2O
 Charge                    0.0000       0.0000  mmol/kgH2O
 Dissolved species     78683.8102   78683.8102    mg/kgH2O
 Density                   1.0329       1.0329    kg/l
-------------------------------------------------------------------

WATER COMPOSITION  (mmol/kgH2O)
-------------------------------------------------------------------
 Ion          Initial              Equilibrium          Act.Coef.
-------------------------------------------------------------------
 H+           1.2650E-01           1.2650E-01           7.8415E-01
 OH-          1.6157E-06           1.6157E-06           5.7961E-01
 Na+          1.3375E+03           1.3375E+03           6.5473E-01
 Cl-          1.3375E+03           1.3375E+03           6.5413E-01
 CO2          1.1650E+01           1.1650E+01           1.1624E+00
 HCO3-        1.2648E-01           1.2648E-01           5.1354E-01
 CO32-        7.2737E-07           7.2737E-07           6.4592E-02
 H2S          1.8316E-03           1.8316E-03           1.1817E+00
 HS-          8.5591E-06           8.5591E-06           5.7490E-01
-------------------------------------------------------------------
  *Only species with concentration > zero are written
```

Figure 4.13 Simulation input data and results for NaCl brine in the presence of 99.995%CO_2/0.005%H_2S.

4.1.7 Repeatability of test

Several factors influence the accuracy of corrosion rate estimation using the LPR method, including kinetic features of the corrosion process, fluid conductivity, the presence of contaminants and corrosion inhibitors added, corroding material properties, measuring sensor quality, and electrode surface treatment. The accuracy or consistency with which the same measurement or experiment may be carried out again under the same circumstances is known as test repeatability. Repeatability is essential for guaranteeing the validity and dependability of experimental results in scientific research and quality control procedures. To identify if the experiments performed were dependable, a bubble test was performed in the presence of 99.99%CO_2 / 0.01% H_2S at pH 4 and 60 °C with phosphate esters, imidazoline and quaternary amines. This experiment was carried out 3 times for individual filming agent and standard deviation was calculated. Figures 4.14, 4.15 and 4.16 shows standard deviation plots for 3 data sets. From the figure we can conclude that the experiments are reliable as every experiment performed quite similarly to their respective parallels. Comparing Figure 4.14 to Figures 4.15 and 4.16, it is evident that the deviation is larger; this could be the consequence of contaminants in the system.

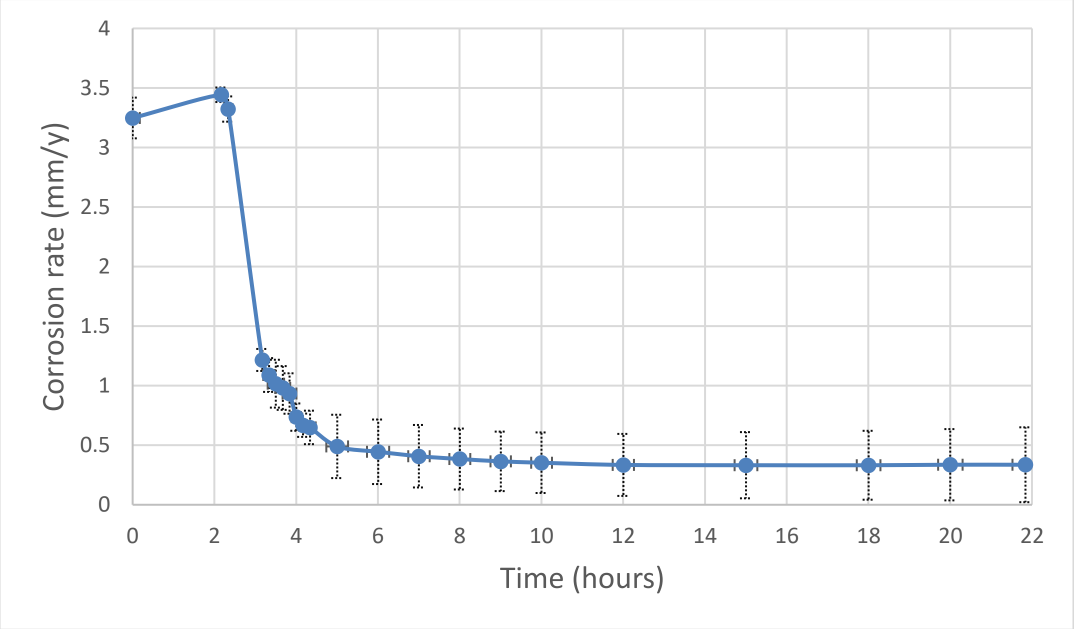


Figure 4.14 Standard deviation of performance of phosphate ester in the presence of 99.99%CO_2/0.01%H_2S gas at pH 4 and 60 °C

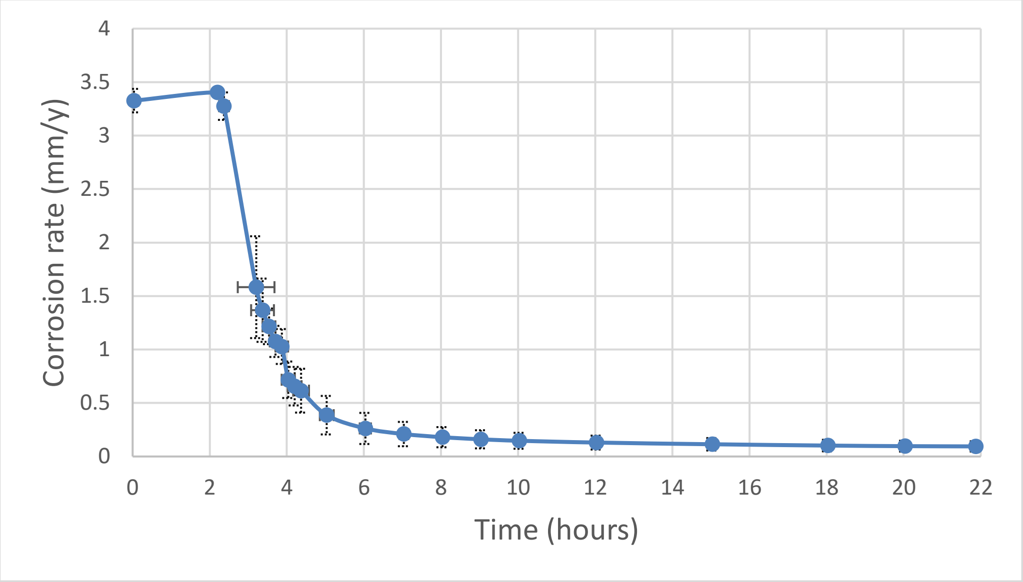


Figure 4.15 Standard deviation of performance of imidazoline in the presence of 99.99%CO_2/0.01%H_2S gas at pH 4 and 60 °C.

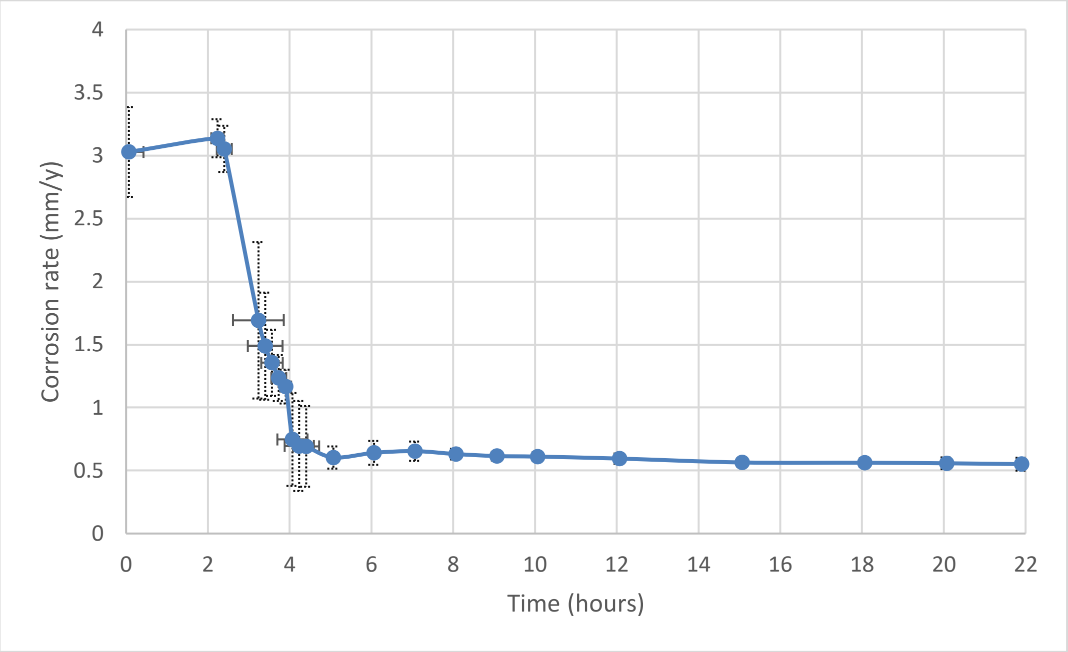


Figure 4.16 Standard deviation of performance of quaternary amines in the presence of 99.99%CO_2/0.01%H_2S gas at pH 4 and 60 °C.

4.2 Corrosion monitoring and inspection with RCA test

The corrosion rate for C1018 subjected to 3% NaCl brine at 60 °C was calculated using weight loss measurements, and the coupons were inspected for signs of local corrosion. Table 4.1 shows the corrosion rate data obtained under various test conditions. The average corrosion rate for imidazoline in the presence of H_2S is 0.3563 mm/y, which is much lower than the corrosion rate in the presence of sulfur compound and pure CO_2, which was reported at 1.084 mm/y. A similar pattern was found with phosphate esters, with an average corrosion rate of 2.0968 mm/y in the presence of pure CO_2 gas, which decreased to 0.6405 mm/y in the presence of CO_2/H_2S gas. It can be concluded that corrosion is more severe in a pure CO_2 atmosphere than in the presence of H_2S. However, a substantial difference in corrosion rate is seen between the two coupons under similar conditions. As a general representation of the corrosion on the coupons, Figures 4.17 and 4.18 display the coupons that were recovered from the testing after a span of 72 hours. A profilometer was then used to analyze the coupons that had local corrosion on the surface, producing 3D pictures and cross-section profiles. The coupons were examined under an optical microscope to see if they contained any pits. Coupons 4877 and 4860 were found to have pits on the backside and were further inspected using a profilometer. Figures 4.19 and 4.20 display the results of the profilometer scans.

Figure 4.17 Coupons after 72 hours immersion in the presence of CO_2.

Figure 4.18 Coupons after 72 hours immersion in the presence of CO_2/H_2S.

Table 4.1 *Effectiveness of corrosion inhibitor with and without sulfur compound*

Filming agent	**Sulfur compound**	**Gas**	**Coupon no.**	**Initial weight**	**Final weight**	**Corrosion rate (mm/y)**	**Local Corrosion**
Phosphate ester	2-Mercaptoethanol	100% CO_2	4876	4.3691	4.2994	1.2417	0
			4877	4.3955	4.2298	2.9519	Pitting on edges
Imidazoline	2-Mercaptoethanol	100% CO_2	4878	4.3383	4.2823	0.9976	0
			4879	4.3866	4.3209	1.1704	0
Phosphate ester	-	99.99% CO_2/0.01% H_2S	4859	4.3747	4.3496	0.4472	0
			4860	4.3599	4.3131	0.8337	Pitting on edges
Imidazoline	-	99.99% CO_2/0.01% H_2S	4882	4.3755	4.3571	0.3278	0
			4883	4.3832	4.3616	0.3848	0
Quaternary amines	-	99.99% CO_2/0.01% H_2S	4856	4.3841	4.3718	0.2191	0
			4857	4.3369	4.3289	0.1425	0

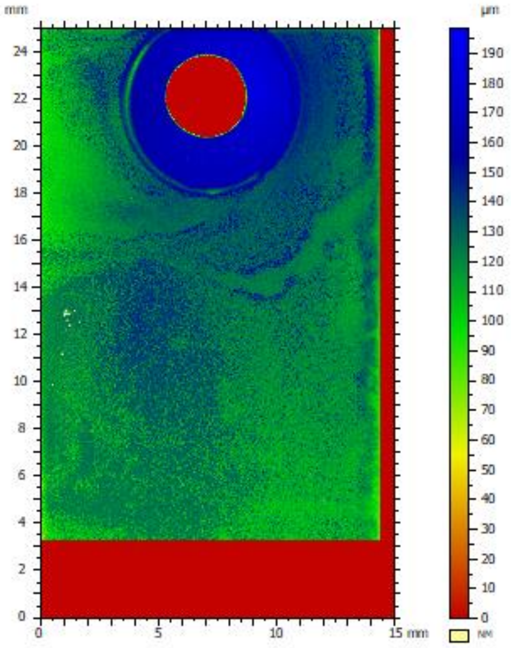


Figure 4.19 Surface profilometry of 4877 coupon after experiment with phosphate ester corrosion inhibitor in pure CO_2.

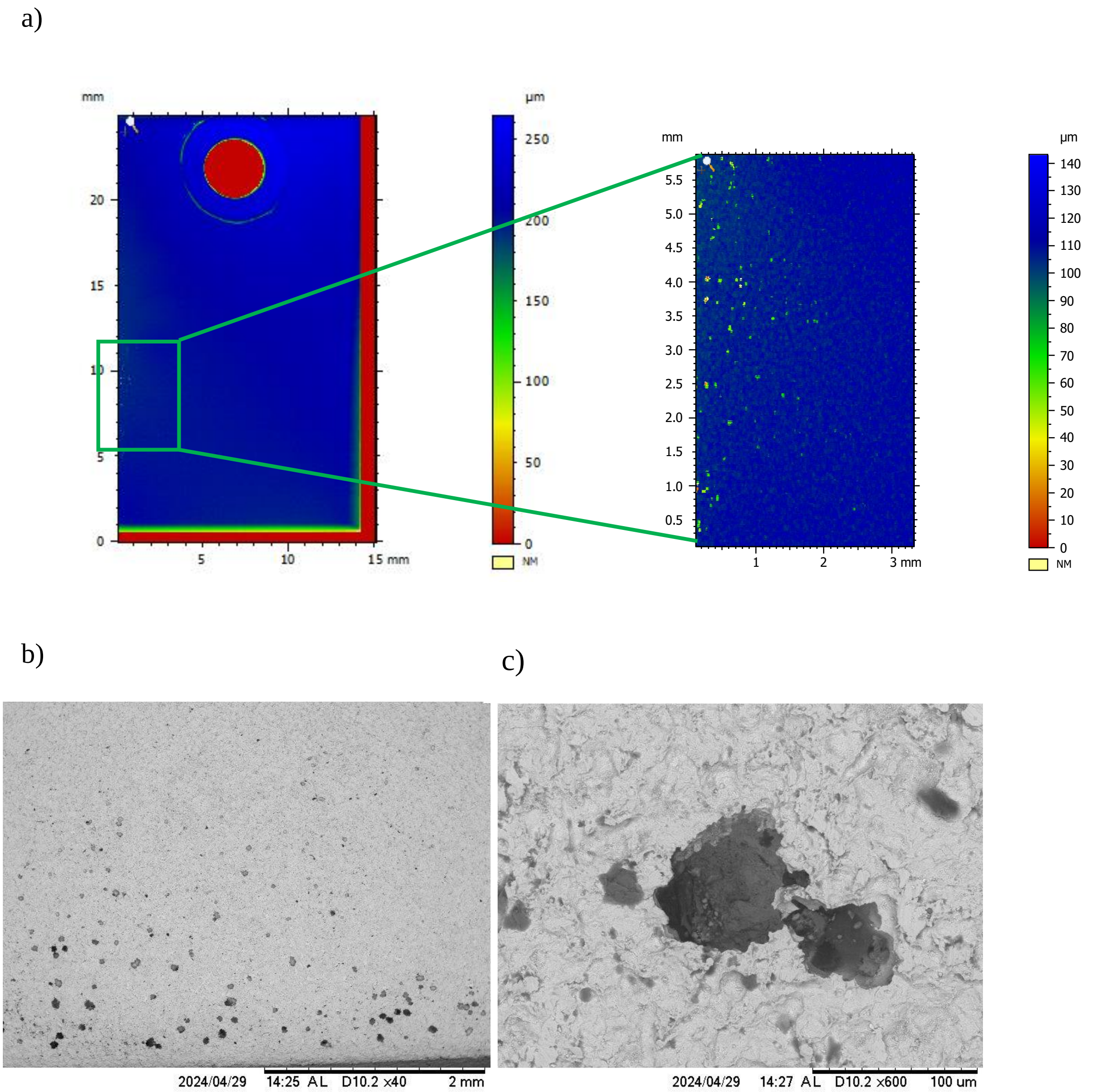
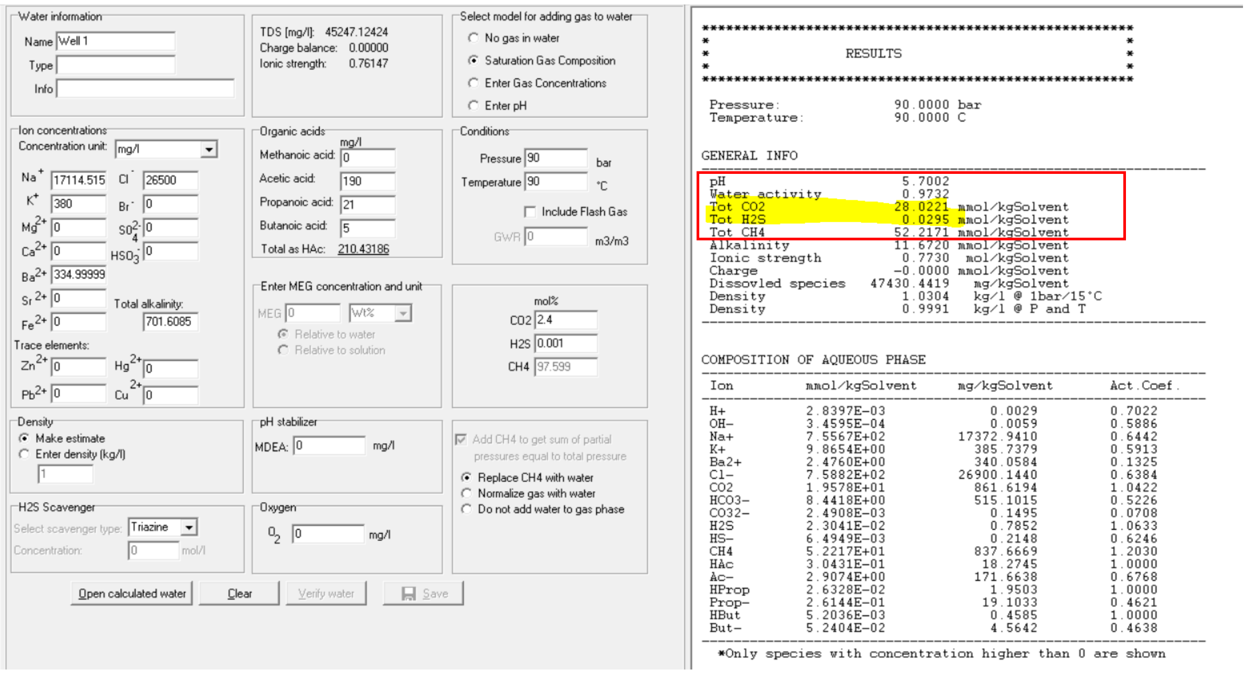


Figure 4.20 a) Surface profilometry of 4860 coupon after experiment with phosphate ester corrosion inhibitor in 99.99%CO_2/0.01%H_2S, b) SEM of 4860 coupon pits (magnification: 40×), c) SEM of 4860 coupon pits (magnification: 600×)

4.3 Field Case Study

In this study, the performance of imidazoline without sulfur compound was evaluated for a field condition. It is vital to understand the behavior of corrosion inhibitor without sulfur compound outside the laboratory conditions for its practical deployment. Corrosion inhibitor performance was evaluated using rotating cage autoclave based on simulated pipeline conditions. The pipeline conditions were simulated using ScaleSim simulation and the conditions were converted into the lab conditions.

The data from the field for a given well with the water analysis from the produced water sample was used as an input to the software.



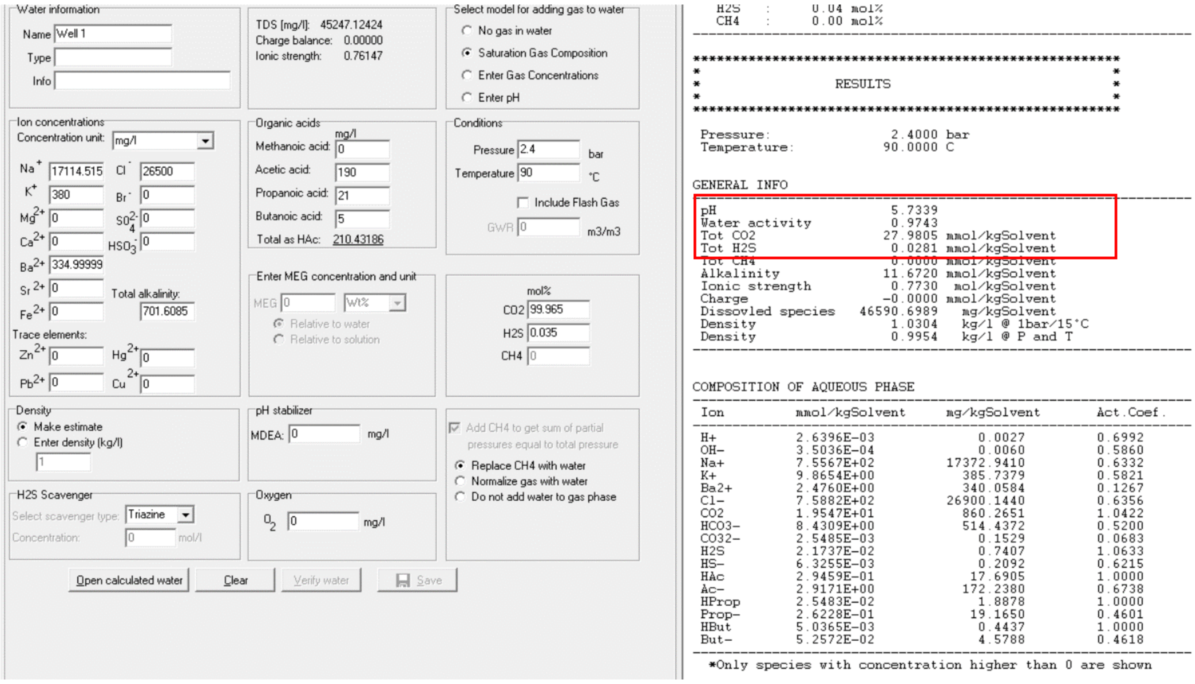
Name: Well 1
Type:
Info:
TDS [mg/l]: 45247.12424
Charge balance: 0.00000
Ionic strength: 0.76147
○ No gas in water
● Saturation Gas Composition
○ Enter Gas Concentrations
○ Enter pH
Concentration unit: mg/l
Na^+ 17114.515 Cl^- 26500
K^+ 380 Br^- 0
Mg^{2+} 0 SO_4^{2-} 0
Ca^{2+} 0 HSO_3^- 0
Ba^{2+} 334.99999
Sr^{2+} 0 Total alkalinity: 701.6085
Fe^{2+} 0
Zn^{2+} 0 Hg^{2+} 0
Pb^{2+} 0 Cu^{2+} 0
Methanoic acid (mg/l): 0
Acetic acid: 190
Propanoic acid: 21
Butanoic acid: 5
Total as HAc: 210.43186
Pressure: 90 bar
Temperature: 90 °C
☐ Include Flash Gas
GWR: 0 m3/m3
MEG: 0 Wt%
● Relative to water
○ Relative to solution
mol%
CO2: 2.4
H2S: 0.001
CH4: 97.599
● Make estimate
○ Enter density (kg/l): 1
MDEA: 0 mg/l
☑ Add CH4 to get sum of partial pressures equal to total pressure
● Replace CH4 with water
○ Normalize gas with water
○ Do not add water to gas phase
Select scavenger type: Triazine
Concentration: 0 mol/l
O_2: 0 mg/l

```
*************************************************************
*                                                           *
*                   RESULTS                                 *
*                                                           *
*************************************************************

 Pressure:                 90.0000 bar
 Temperature:              90.0000 C


GENERAL INFO
----------------------------------------------------------------
 pH                         5.7002
 Water activity             0.9732
 Tot CO2                   28.0221 mmol/kgSolvent
 Tot H2S                    0.0295 mmol/kgSolvent
 Tot CH4                   52.2171 mmol/kgSolvent
 Alkalinity                11.6720 mmol/kgSolvent
 Ionic strength             0.7730  mol/kgSolvent
 Charge                    -0.0000 mmol/kgSolvent
 Dissovled species      47430.4419   mg/kgSolvent
 Density                    1.0304   kg/l @ 1bar/15°C
 Density                    0.9991   kg/l @ P and T
----------------------------------------------------------------


COMPOSITION OF AQUEOUS PHASE
----------------------------------------------------------------
 Ion         mmol/kgSolvent     mg/kgSolvent        Act.Coef.
----------------------------------------------------------------
 H+          2.8397E-03             0.0029          0.7022
 OH-         3.4595E-04             0.0059          0.5886
 Na+         7.5567E+02         17372.9410          0.6442
 K+          9.8654E+00           385.7379          0.5913
 Ba2+        2.4760E+00           340.0584          0.1325
 Cl-         7.5882E+02         26900.1440          0.6384
 CO2         1.9578E+01           861.6194          1.0422
 HCO3-       8.4418E+00           515.1015          0.5226
 CO32-       2.4908E-03             0.1495          0.0708
 H2S         2.3041E-02             0.7852          1.0633
 HS-         6.4949E-03             0.2148          0.6246
 CH4         5.2217E+01           837.6669          1.2030
 HAc         3.0431E-01            18.2745          1.0000
 Ac-         2.9074E+00           171.6638          0.6768
 HProp       2.6328E-02             1.9503          1.0000
 Prop-       2.6144E-01            19.1033          0.4621
 HBut        5.2036E-03             0.4585          1.0000
 But-        5.2404E-02             4.5642          0.4638
----------------------------------------------------------------
  *Only species with concentration higher than 0 are shown
```

Figure 4.21 Simulation results for field conditions.

The most important factors for evaluating corrosion are pH and total CO_2 in aqueous [38], which served as the foundation for converting to lab conditions. A 99.965% CO_2/0.0365% H_2S gas is utilized in the lab, and the total pressure was adjusted to fit either the concentration of CO_2 and H_2S in the aqueous phase or a corresponding partial pressure of CO_2 and H_2S in the field. Since the total CO_2 in the liquid takes into account the pertinent fugacity coefficients and is directly correlated with the CO_2 corrosion rate, it will be more accurate than the partial pressure.

NORSOK M-506 corrosion model was then used to simulate the pipeline conditions and calculate shear stress. Based on the obtained shear stress i.e. 1Pa, the flow in the pipe is laminar and then the rotation speed of the autoclave was calculated using the equation 2.2 and 2.3. The brine composition and test conditions for the specific field are summarized in Table 4.2 and 4.3.



Name: Well 1
Type:
Info:

TDS [mg/l]: 45247.12424
Charge balance: 0.00000
Ionic strength: 0.76147

○ No gas in water
● Saturation Gas Composition
○ Enter Gas Concentrations
○ Enter pH

Concentration unit: mg/l

Na^+: 17114.515
Cl^-: 26500
K^+: 380
Br^-: 0
Mg^{2+}: 0
SO_4^{2-}: 0
Ca^{2+}: 0
HSO_3^-: 0
Ba^{2+}: 334.99999
Sr^{2+}: 0
Total alkalinity: 701.6085
Fe^{2+}: 0
Zn^{2+}: 0
Hg^{2+}: 0
Pb^{2+}: 0
Cu^{2+}: 0

Methanoic acid (mg/l): 0
Acetic acid: 190
Propanoic acid: 21
Butanoic acid: 5
Total as HAc: 210.43186

MEG: 0 Wt%
● Relative to water
○ Relative to solution

Pressure: 2.4 bar
Temperature: 90 °C
☐ Include Flash Gas
GWR: 0 m3/m3

mol%
CO2: 99.965
H2S: 0.035
CH4: 0

● Make estimate
○ Enter density (kg/l)
1

MDEA: 0 mg/l

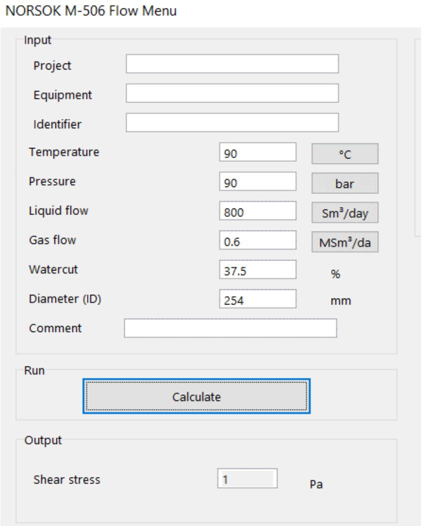
☑ Add CH4 to get sum of partial pressures equal to total pressure
● Replace CH4 with water
○ Normalize gas with water
○ Do not add water to gas phase

Select scavenger type: Triazine
Concentration: 0 mol/l

O_2: 0 mg/l

```
   H2S    :      0.04 mol%
   CH4    :      0.00 mol%
--------------------------------------------------------------

*******************************************************
*                                                     *
*                  RESULTS                            *
*                                                     *
*******************************************************

 Pressure:                   2.4000 bar
 Temperature:               90.0000 C

GENERAL INFO
--------------------------------------------------------------
 pH                          5.7339
 Water activity              0.9743
 Tot CO2                    27.9805 mmol/kgSolvent
 Tot H2S                     0.0281 mmol/kgSolvent
 Tot CH4                     0.0000 mmol/kgSolvent
 Alkalinity                 11.6720 mmol/kgSolvent
 Ionic strength              0.7730  mol/kgSolvent
 Charge                     -0.0000 mmol/kgSolvent
 Dissovled species       46590.6989   mg/kgSolvent
 Density                     1.0304   kg/l @ 1bar/15°C
 Density                     0.9954   kg/l @ P and T
--------------------------------------------------------------


COMPOSITION OF AQUEOUS PHASE
--------------------------------------------------------------
 Ion          mmol/kgSolvent      mg/kgSolvent        Act.Coef.
--------------------------------------------------------------
 H+           2.6396E-03            0.0027            0.6992
 OH-          3.5036E-04            0.0060            0.5860
 Na+          7.5567E+02        17372.9410            0.6332
 K+           9.8654E+00          385.7379            0.5821
 Ba2+         2.4760E+00          340.0584            0.1267
 Cl-          7.5882E+02        26900.1440            0.6356
 CO2          1.9547E+01          860.2651            1.0422
 HCO3-        8.4309E+00          514.4372            0.5200
 CO32-        2.5485E-03            0.1529            0.0683
 H2S          2.1737E-02            0.7407            1.0633
 HS-          6.3255E-03            0.2092            0.6215
 HAc          2.9459E-01           17.6905            1.0000
 Ac-          2.9171E+00          172.2380            0.6738
 HProp        2.5483E-02            1.8878            1.0000
 Prop-        2.6228E-01           19.1650            0.4601
 HBut         5.0365E-03            0.4437            1.0000
 But-         5.2572E-02            4.5788            0.4618
--------------------------------------------------------------
  *Only species with concentration higher than 0 are shown
```

Figure 4.22 Simulation results for lab conditions.



Project:
Equipment:
Identifier:
Temperature: 90 °C
Pressure: 90 bar
Liquid flow: 800 Sm³/day
Gas flow: 0.6 MSm³/da
Watercut: 37.5 %
Diameter (ID): 254 mm
Comment:

Shear stress: 1 Pa

Figure 4.23 Results from the NORSOK software.

Table 4.2 Brine composition

Salt	**Weight (mg/l)**
KCl	0.725
$BaCl_2.2H_2O$	0.596
NaCl	42.833
$NaHCO_3$	0.736

Table 4.3 Test conditions for rotating cage autoclave experiment

pH	5.7
Temperature, °C	90
Time, hours	72
Pressure, bar	2.4
CO_2/H_2S gas	99.965%/0.035%
CO_2, ml/min	85.7
H_2S, ml/min	50
Shear stress, Pa	1
Rotation, rpm	145
Sodium Acetate, mg/l	287
Filming agent (FA)	Imidazoline
FA dose	30 ppm

Table 4.4 presents the findings from the rotating cage autoclave test. The average corrosion rate of imidazoline corrosion inhibitor in the presence of 0.035% H_2S is 0.0365 mm/y, which is lower than the rate found in the presence of both sulfur compound and 0.035% H_2S, which was 0.08 mm/y. The coupons that were recovered from the test after 72 hours are shown in Figure 4.24. Upon examination of the coupons using an optical microscope, no indication of localized corrosion was found. Nonetheless, it can be seen from the profilometry scans in Figures 4.25 and 4.26 that the 4892 coupon has higher degrees of corrosion than the 4892 coupon. However, it is advisable to run parallels for this test to strongly support the findings.

Table 4.4 Effectiveness of Imidazoline with and without sulfur compound

Filming agent	**Sulfur compound**	**Gas**	**Coupon no.**	**Initial weight**	**Final weight**	**Corrosion rate (mm/y)**	**Local Corrosion**
Imidazoline	2-Mercaptoethanol	99.965% CO_2/0.035% H_2S	4892	4.4176	4.4121	0.098	No pits
			4893	4.385	4.3815	0.062	No pits
Imidazoline	-	99.965% CO_2/0.035% H_2S	4894	4.375	4.3728	0.039	No pits
			4895	4.3839	4.382	0.034	No pits

Figure 4.24 Coupons after 72 hours immersion in the presence of 99.965% CO_2 / 0.035% H_2S.

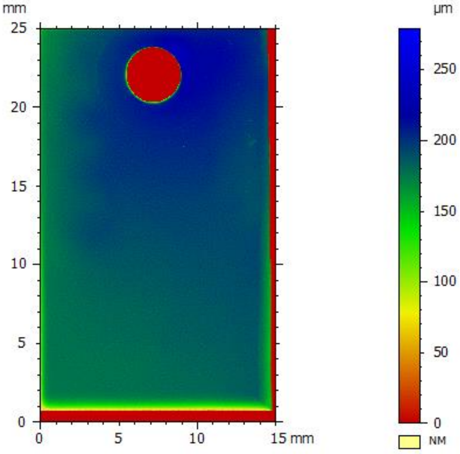


Figure 4.25 Surface profilometry of 4892 coupon.

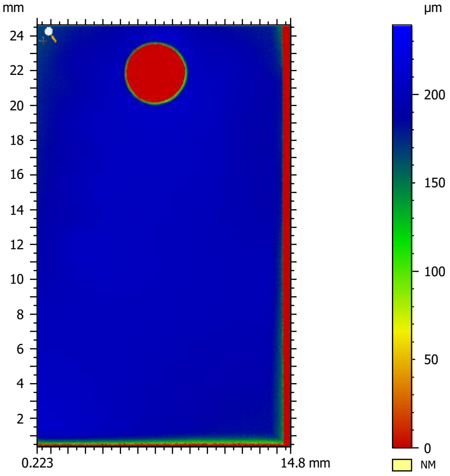


Figure 4.26 Surface profilometry of 4894 coupon.

5 Conclusion

The objective of this study is to remove the sulfur ingredient from the corrosion inhibitor formulation and examine the synergistic impact of H_2S in the absence of the sulfur compound on C1018 carbon steel. Based on the comprehensive results obtained from the corrosion inhibition experiments, it is evident that the presence of H_2S significantly impacts the performance of various corrosion inhibitors under different test conditions. Here's a concise conclusion summarizing the key findings:

- The tests examined whether sulfur compounds are required in field applications to achieve efficient corrosion inhibition by comparing laboratory and field settings. The outcomes of corrosion monitoring techniques, including weight loss calculations, rotating cage autoclave, and bubble tests, provide valuable insights into the performance of corrosion inhibitors in different conditions. Phosphate esters, imidazoline, and quaternary amines showed higher inhibition efficiency in CO_2/H_2S environments without sulfur compounds. This indicates that in the presence of H_2S gas, filming agents formed stronger bonds with metal surfaces, improving corrosion protection.

- The influence of pH, temperature, and H_2S concentration on corrosion inhibitor performance was also explored in the presence of H_2S and without sulfur compound. Higher pH levels were found to enhance corrosion protection. Increased temperatures greatly reduced quaternary amines adsorption while only marginally limiting phosphate esters and imidazoline adsorption. Furthermore, the synergistic effect of H_2S on corrosion inhibitors demonstrated the need to comprehend the complex chemical interactions in corrosion environments. Interestingly, even at lower molar concentrations of H_2S, imidazolines and quaternary amines demonstrated good performance with similar inhibitory efficiency. Imidazolines showed 97% and 92% inhibitory rates in 0.01% and 0.005% H_2S, respectively, and quaternary amines exhibited a similar stability. On the other hand, phosphate esters performed better in the presence of 0.01% H_2S as compared to 0.005% H_2S.

- The repeatability of tests confirmed the reliability of experimental results, essential for ensuring the validity of corrosion inhibition studies.

- The field case study focused on evaluating the effectiveness of an imidazoline corrosion inhibitor in the presence of $99.965\%CO_2/0.035\%H_2S$, both with and without 2-mercaptoethanol. The corrosion rate of 0.0365 mm/y, observed when 0.035% H_2S was present, demonstrates the efficacy of H_2S in reducing corrosion. In contrast, the corrosion rate increased to 0.08 mm/y when a sulfur compound was also present. The lack of localized corrosion on the retrieved coupons verified the inhibitor's effectiveness in providing protection.

In conclusion, the findings highlight the critical role of H_2S as synergists in corrosion inhibition, offering useful insights for optimizing inhibitor formulations. Elimination of sulfur compound from corrosion inhibitor formulations will also contribute towards better environmental impact and will mitigate the health risks associated with handling sulfur compounds for the workers.

6 Recommendations for future work

The conducted experiments yielded satisfactory results considering the project's scope. Future research is still needed, nevertheless, to examine corrosion inhibitors using the rotating cage autoclave experiment in the presence of H_2S under various test conditions, such as varying the temperature, shear rate, and H_2S concentration. It is also noted for future effort to identify the corrosion film's chemistry in order to comprehend its composition and morphology. It would be interesting to examine the nature of the sulfur film produced as there are various polymorphs of iron sulfide. The formulation and evaluation of corrosion inhibitors in the absence of sulfur compounds in a CO_2/H_2S environment should be investigated for various real-field conditions. Finally, in order to strongly corroborate the findings in this study, it is recommended to do parallel experiments for each of them.

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Appendices

Appendix A: Project Description

Appendix B: Test Matrix

Appendix C: Chemical Inforcard retrieved from European Chemicals Agency (ECHA)

Appendix A

Title: Experimental investigation of sulphur compound elimination from corrosion inhibitor

USN supervisor: Per Morten Hansen

External partner: Schlumberger (SLB) Tore Nordvik

Task background:
Corrosion inhibitors are widely used in the oil and gas industry to protect metal surfaces from corrosion. However, film-forming surfactant components may exhibit varying degrees of corrosion protection in real-world conditions compared to controlled laboratory tests. As a result, in addition to the filming agents and the solvent package, a sulfur-containing ingredient is frequently added to the corrosion inhibitor formulation. These sulfur additives interact with iron to generate intricate compounds capable of forming protective coatings and enhancing surfactant adhesion to metal surfaces. Nonetheless, the influence of sulfur ingredients on the performance of corrosion inhibitors under actual field conditions remains a subject of ongoing investigation. Some filming corrosion inhibitors were observed to work successfully without sulfur compound in the field. But when the same corrosion inhibitor was tested in the laboratory setup, it did not perform.

Task description:
The project's main objective is to eliminate sulphur compound from the corrosion inhibitor. In this project, following tasks will be performed:

- Literature study. Identify the crucial factors for the adsorption mechanism.
- Investigate the function of H2S as a sulphur compound.
- Develop method to replicate the field conditions in a laboratory setting using H2S.
- Evaluate the method for replication of field conditions using H2S.
- Evaluate the inhibition efficiency of corrosion inhibitors with and without sulfur compound in presence of H2S.
- Carry out Bubble testing and linear polarization resistance (LPR) measurement to evaluate the corrosion rate.
- Compare the results of corrosion inhibitors with and without sulfur compound.
- Evaluate the results and identify uncertainties.

Student category: The thesis is allocated to EET student Aditi Santosh Mhatre

Is the task suitable for online students (not present at the campus)? Yes

Practical arrangements: Most supervision meetings with USN will be on TEAMS.

Supervision:
As a general rule, the student is entitled to 15-20 hours of supervision. This includes necessary time for the supervisor to prepare for supervision meetings (reading material to be discussed, etc).

Signatures:

Supervisor (date and signature): 1.2.2024 Per Morten Hansen

Student (write clearly in all capitalized letters): ADITI SANTOSH MHATRE

Student (date and signature): 1.5.2024

Appendix B

Im – Imidazoline

PE – Phosphate ester

QAS – Quaternary ammonium salts

TS – Thiosulfate

TGA – Thioglyclic acid

ME – 2-Mercaptoethanol

Table B.1 Test matrix for bubble test

Test No	**Gas**	**Gas Composition**	**pH**	**Temp ©**	**Filming agent + Sulfur compound added**			
					Kettle 1	**Kettle 2**	**Kettle 3**	**Kettle 4**
1	CO_2	100%	4	60	PE	PE + TS	PE + TGA	PE + ME
2	CO_2	100%	4	60	Im	Im + TS	Im + TGA	Im + ME
3	CO_2	100%	4	60	QAS	QAS + TS	QAS + TGA	QAS + ME
4	CO_2/H_2S	99.99%/0.01%	4	60	PE	Im	QAS	-
5	CO_2/H_2S	99.995%/0.005%	4	60	PE	Im	QAS	-
6	CO_2/H_2S	99.99%/0.01%	4	20	PE	Im	QAS	-
7	CO_2/H_2S	99.99%/0.01%	6	60	PE	Im	QAS	-
8	CO_2/H_2S	99.99%/0.01%	6	20	PE	Im	QAS	-

Table B.2 Test matrix for rotating cage autoclave test

Test no	**Gas**	**Gas Composition**	**pH**	**Temp ©**	**Pressure (barg)**	**Shear rate (rpm)**	**Time (hrs)**	**Kettle volume (ml)**	**Chemical (FA +SC)**	**Coupon ID**
1	CO2	100%	4	60	0.0	200	72	750	PE + ME	4876
										4877
2	CO2	100%	4	60	0.0	200	72	750	Im + ME	4878
										4879
3	CO2/H2S	99.99%/0.01%	4	60	0.0	200	72	750	PE	4859
										4860
4	CO2/H2S	99.99%/0.01%	4	60	0.0	200	72	750	Im	4882
										4883
5	CO2/H2S	99.99%/0.01%	4	60	0.0	200	72	750	QAS	4856
										4857
6	CO2/H2S	99.965%/0.035%	5.7	90	2.4	145	72	750	Im + ME	4892
										4893
7	CO2/H2S	99.965%/0.035%	5.7	90	2.4	145	72	750	Im	4894
										4895

Tall oil, reaction products with diethylenetriamine

Substance identity

EC / List no.: 268-776-5

CAS no.: 68140-14-7

Mol. formula:

No image available

Hazard classification & labelling

Danger! According to the classification provided by companies to ECHA in **REACH registrations** this substance causes severe skin burns and eye damage, is very toxic to aquatic life, is very toxic to aquatic life with long lasting effects and may cause an allergic skin reaction.

Properties of concern

Ss A majority of data submitters agree this substance is Skin sensitising

About this substance

This substance is registered under the REACH Regulation and is manufactured in and / or imported to the European Economic Area, at ≥ 10 to < 100 tonnes per annum.

This substance is used by professional workers (widespread uses), in formulation or re-packing, at industrial sites and in manufacturing.

This substance is used by professional workers (widespread uses), in formulation or re-packing, at industrial sites and in manufacturing.

Consumer Uses

ECHA has no public registered data indicating whether or in which chemical products the substance might be used. ECHA has no public registered data on the routes by which this substance is most likely to be released to the environment.

Article service life

ECHA has no public registered data on the routes by which this substance is most likely to be released to the environment. ECHA has no public registered data indicating whether or into which articles the substance might have been processed.

Widespread uses by professional workers

ECHA has no public registered data indicating whether or in which chemical products the substance might be used. ECHA has no public registered data on the types of manufacture using this substance. Other release to the environment of this substance is likely to occur from: indoor use as reactive substance.

Formulation or re-packing

ECHA has no public registered data indicating whether or in which chemical products the substance might be used. Release to the environment of this substance can occur from industrial use: formulation of mixtures.

Uses at industrial sites

ECHA has no public registered data indicating whether or in which chemical products the substance might be used. This substance is used in the following areas: offshore mining.
Release to the environment of this substance can occur from industrial use: in processing aids at industrial sites, as an intermediate step in further manufacturing of another substance (use of intermediates) and as processing aid.

Manufacture

Release to the environment of this substance can occur from industrial use: manufacturing of the substance.

about INFOCARD - Last updated: 06/05/2022

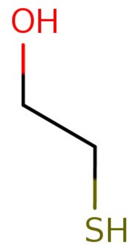
2-mercaptoethanol

Substance identity

EC / List no.: 200-464-6

CAS no.: 60-24-2

Mol. formula: C2H6OS



Hazard classification & labelling

Danger! According to the classification provided by companies to ECHA in **REACH registrations** this substance is fatal in contact with skin, is toxic if swallowed, is toxic if inhaled, is very toxic to aquatic life, is toxic to aquatic life with long lasting effects, causes serious eye damage, is suspected of damaging fertility or the unborn child, may cause damage to organs through prolonged or repeated exposure, causes skin irritation and may cause an allergic skin reaction.

Properties of concern

Ss A majority of data submitters agree this substance is Skin sensitising

About this substance

This substance is registered under the REACH Regulation and is manufactured in and / or imported to the European Economic Area, at ≥ 1 000 to < 10 000 tonnes per annum.

This substance is used by professional workers (widespread uses), in formulation or re-packing, at industrial sites and in manufacturing.

This substance is used by professional workers (widespread uses), in formulation or re-packing, at industrial sites and in manufacturing.

Consumer Uses

ECHA has no public registered data indicating whether or in which chemical products the substance might be used. ECHA has no public registered data on the routes by which this substance is most likely to be released to the environment.

Article service life

ECHA has no public registered data on the routes by which this substance is most likely to be released to the environment. ECHA has no public registered data indicating whether or into which articles the substance might have been processed.

Widespread uses by professional workers

This substance is used in the following products: pH regulators and water treatment products and laboratory chemicals.
This substance is used in the following areas: health services and scientific research and development.
Other release to the environment of this substance is likely to occur from: indoor use (e.g. machine wash liquids/detergents, automotive care products, paints and coating or adhesives, fragrances and air fresheners).

Formulation or re-packing

This substance is used in the following products: pH regulators and water treatment products.
Release to the environment of this substance can occur from industrial use: formulation of mixtures.

Uses at industrial sites

This substance is used in the following products: pH regulators and water treatment products, metal surface treatment products and water treatment chemicals.
This substance has an industrial use resulting in manufacture of another substance (use of intermediates).
This substance is used in the following areas: mining and formulation of mixtures and/or re-packaging.
This substance is used for the manufacture of: chemicals, and metals.
Release to the environment of this substance can occur from industrial use: in processing aids at industrial sites, as an intermediate step in further manufacturing of another substance (use of intermediates), as processing aid and as processing aid.

Manufacture

Release to the environment of this substance can occur from industrial use: manufacturing of the substance.

ECHA
EUROPEAN CHEMICALS AGENCY

about INFOCARD - Last updated: 06/09/2023

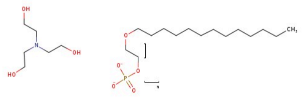
Ethanol, 2,2',2''-nitrilotris-, compd. with α-tridecyl-.omega.- hydroxypoly(oxy-1,2-ethanediyl) phosphate

Substance identity

EC / List no.: 630-538-7

CAS no.: 58855-61-1

Mol. formula:



Hazard classification & labelling

Danger! According to the classification provided by companies to ECHA in **CLP notifications** this substance is toxic to aquatic life with long lasting effects, causes serious eye damage and causes skin irritation.

about INFOCARD - Last updated: 06/09/2023

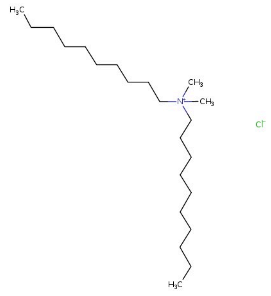
Didecyldimethylammonium chloride

Substance identity

EC / List no.: 230-525-2

CAS no.: 7173-51-5

Mol. formula: C22H48ClN



Hazard classification & labelling

Danger! According to the **harmonised classification and labelling** (CLP00) approved by the European Union, this substance causes severe skin burns and eye damage and is harmful if swallowed.

Additionally, the classification provided by companies to ECHA in **REACH registrations** identifies that this substance is toxic if swallowed, is very toxic to aquatic life, is toxic to aquatic life with long lasting effects, causes serious eye damage, is a flammable liquid and vapour and may cause drowsiness or dizziness.

About this substance

This substance is registered under the REACH Regulation and is manufactured in and / or imported to the European Economic Area, at ≥ 100 to < 1 000 tonnes per annum.

This substance is used by consumers, by professional workers (widespread uses), in formulation or re-packing, at industrial sites and in manufacturing.

Biocidal Uses

This substance is approved for use as a biocide in the EEA and/or Switzerland, for: human hygiene, disinfection, veterinary hygiene, food and animals feeds, wood preservation.

This substance is being reviewed for use as a biocide in the EEA and/or Switzerland, for: product preservation, preservation for construction materials, preservation for liquid systems, controlling slimes.

This substance is used by consumers, by professional workers (widespread uses), in formulation or re-packing, at industrial sites and in manufacturing.

Consumer Uses

This substance is used in the following products: biocides (e.g. disinfectants, pest control products), washing & cleaning products, plant protection products and cosmetics and personal care products.
Other release to the environment of this substance is likely to occur from: indoor use as processing aid and outdoor use as processing aid.

Article service life

ECHA has no public registered data on the routes by which this substance is most likely to be released to the environment. ECHA has no public registered data indicating whether or into which articles the substance might have been processed.

Widespread uses by professional workers

This substance is used in the following products: biocides (e.g. disinfectants, pest control products), washing & cleaning products, plant protection products and water treatment chemicals.
This substance is used in the following areas: mining and health services.
This substance is used for the manufacture of: chemicals.
Other release to the environment of this substance is likely to occur from: indoor use (e.g. machine wash liquids/detergents, automotive care products, paints and coating or adhesives, fragrances and air fresheners) and outdoor use.

Formulation or re-packing

This substance is used in the following products: biocides (e.g. disinfectants, pest control products), washing & cleaning products, water treatment chemicals, polishes and waxes, pH regulators and water treatment products, air care products, paper chemicals and dyes and cosmetics and personal care products.
Release to the environment of this substance can occur from industrial use: formulation of mixtures.

Uses at industrial sites

This substance is used in the following products: biocides (e.g. disinfectants, pest control products), washing & cleaning products, plant protection products, water treatment chemicals and oil and gas exploration or production products.
This substance is used in the following areas: mining.
This substance is used for the manufacture of: chemicals and pulp, paper and paper products.
Release to the environment of this substance can occur from industrial use: in processing aids at industrial sites, as processing aid and of substances in closed systems with minimal release.

Manufacture

Release to the environment of this substance can occur from industrial use: manufacturing of the substance.

about INFOCARD - Last updated: 06/09/2023

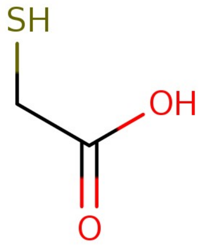
Mercaptoacetic acid

Substance identity

EC / List no.: 200-677-4

CAS no.: 68-11-1

Mol. formula: $C_2H_4O_2S$



Hazard classification & labelling

Danger! According to the **harmonised classification and labelling** (CLP00) approved by the European Union, this substance is toxic if swallowed, is toxic in contact with skin, causes severe skin burns and eye damage and is toxic if inhaled.

Additionally, the classification provided by companies to ECHA in **REACH registrations** identifies that this substance causes serious eye damage, is harmful to aquatic life with long lasting effects and may cause an allergic skin reaction.

Properties of concern

Ss Some data submitters indicate they consider this substance as Skin sensitising

About this substance

This substance is registered under the REACH Regulation and is manufactured in and / or imported to the European Economic Area, at ≥ 10 000 to < 100 000 tonnes per annum.

This substance is used by consumers, by professional workers (widespread uses), in formulation or re-packing, at industrial sites and in manufacturing.

This substance is used by consumers, by professional workers (widespread uses), in formulation or re-packing, at industrial sites and in manufacturing.

Consumer Uses

This substance is used in the following products: cosmetics and personal care products.
Other release to the environment of this substance is likely to occur from: indoor use as processing aid.

Article service life

ECHA has no public registered data on the routes by which this substance is most likely to be released to the environment. ECHA has no public registered data indicating whether or into which articles the substance might have been processed.

Widespread uses by professional workers

This substance is used in the following products: laboratory chemicals and cosmetics and personal care products.
This substance is used in the following areas: scientific research and development.
Other release to the environment of this substance is likely to occur from: indoor use as reactive substance and indoor use in close systems with minimal release (e.g. cooling liquids in refrigerators, oil-based electric heaters).

Formulation or re-packing

This substance is used in the following products: leather treatment products and cosmetics and personal care products.
This substance has an industrial use resulting in manufacture of another substance (use of intermediates).
Release to the environment of this substance can occur from industrial use: formulation of mixtures.

Uses at industrial sites

This substance is used in the following products: oil and gas exploration or production products.
This substance has an industrial use resulting in manufacture of another substance (use of intermediates).
This substance is used in the following areas: mining and scientific research and development.
This substance is used for the manufacture of: chemicals.
Release to the environment of this substance can occur from industrial use: of substances in closed systems with minimal release, in processing aids at industrial sites and as an intermediate step in further manufacturing of another substance (use of intermediates).

Manufacture

Release to the environment of this substance can occur from industrial use: manufacturing of the substance.

about INFOCARD - Last updated: 06/09/2023

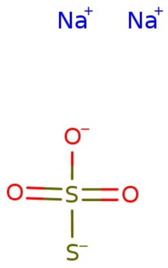
Sodium thiosulphate

Substance identity

EC / List no.: 231-867-5

CAS no.: 7772-98-7

Mol. formula: $Na_2O_3S_2$



Hazard classification & labelling

According to the notifications provided by companies to ECHA in REACH registrations no hazards have been classified.

About this substance

This substance is registered under the REACH Regulation and is manufactured in and / or imported to the European Economic Area, at ≥ 1 000 to < 10 000 tonnes per annum.

This substance is used by consumers, in articles, by professional workers (widespread uses), in formulation or re-packing, at industrial sites and in manufacturing.

This substance is used by consumers, in articles, by professional workers (widespread uses), in formulation or re-packing, at industrial sites and in manufacturing.

Consumer Uses

This substance is used in the following products: fillers, putties, plasters, modelling clay, photo-chemicals, pharmaceuticals, textile treatment products and dyes and cosmetics and personal care products.
Other release to the environment of this substance is likely to occur from: indoor use (e.g. machine wash liquids/detergents, automotive care products, paints and coating or adhesives, fragrances and air fresheners), outdoor use and outdoor use in long-life materials with low release rate (e.g. metal, wooden and plastic construction and building materials).

Article service life

Other release to the environment of this substance is likely to occur from: outdoor use in long-life materials with low release rate (e.g. metal, wooden and plastic construction and building materials) and indoor use in long-life materials with low release rate (e.g. flooring, furniture, toys, construction materials, curtains, foot-wear, leather products, paper and cardboard products, electronic equipment).
This substance can be found in products with material based on: stone, plaster, cement, glass or ceramic (e.g. dishes, pots/pans, food storage containers, construction and isolation material), fabrics, textiles and apparel (e.g. clothing, mattress, curtains or carpets, textile toys), leather (e.g. gloves, shoes, purses, furniture), fabrics, textiles and apparel used for articles with intense direct dermal (skin) contact during normal use (e.g. clothing, shirts, pants, shorts) and leather used for articles with intense direct dermal (skin) contact during normal use (e.g. clothing such as jackets, shoes, or gloves).

Widespread uses by professional workers

This substance is used in the following products: washing & cleaning products, metal surface treatment products, pH regulators and water treatment products, photo-chemicals, water treatment chemicals, fillers, putties, plasters, modelling clay, leather treatment products, polishes and waxes and textile treatment products and dyes.
This substance is used in the following areas: mining, printing and recorded media reproduction, health services, municipal supply (e.g. electricity, steam, gas, water) and sewage treatment, scientific research and development and building & construction work.
This substance is used for the manufacture of: fabricated metal products, textile, leather or fur, pulp, paper and paper products and mineral products (e.g. plasters, cement).
Release to the environment of this substance can occur from industrial use: formulation of mixtures, in processing aids at industrial sites, as processing aid, formulation in materials, in the production of articles, manufacturing of the substance and of substances in closed systems with minimal release.
Other release to the environment of this substance is likely to occur from: indoor use (e.g. machine wash liquids/detergents, automotive care products, paints and coating or adhesives, fragrances and air fresheners) and outdoor use.

Formulation or re-packing

This substance has an industrial use resulting in manufacture of another substance (use of intermediates).
Release to the environment of this substance can occur from industrial use: formulation of mixtures, formulation in materials, in processing aids at industrial sites, as processing aid, in the production of articles, of substances in closed systems with minimal release and manufacturing of the substance.
Other release to the environment of this substance is likely to occur from: indoor use (e.g. machine wash liquids/detergents, automotive care products, paints and coating or adhesives, fragrances and air fresheners) and outdoor use.

Uses at industrial sites

This substance has an industrial use resulting in manufacture of another substance (use of intermediates).
This substance is used in the following areas: formulation of mixtures and/or re-packaging, mining, printing and recorded media reproduction, health services, building & construction work and municipal supply (e.g. electricity, steam, gas, water) and sewage treatment.
This substance is used for the manufacture of: chemicals, textile, leather or fur, pulp, paper and paper products, mineral products (e.g. plasters, cement) and fabricated metal products.
Release to the environment of this substance can occur from industrial use: formulation of mixtures, as processing aid, in processing aids at industrial sites, in the production of articles, formulation in materials, of substances in closed systems with minimal release and manufacturing of the substance.
Other release to the environment of this substance is likely to occur from: indoor use (e.g. machine wash liquids/detergents, automotive care products, paints and coating or adhesives, fragrances and air fresheners) and outdoor use.

Manufacture

Release to the environment of this substance can occur from industrial use: manufacturing of the substance, formulation of mixtures, formulation in materials, in processing aids at industrial sites, in the production of articles, as processing aid, as an intermediate step in

further manufacturing of another substance (use of intermediates) and of substances in closed systems with minimal release.
Other release to the environment of this substance is likely to occur from: indoor use (e.g. machine wash liquids/detergents, automotive care products, paints and coating or adhesives, fragrances and air fresheners).