Nitrogen-containing five-membered heterocyclic compounds as corrosion inhibitors for metals in solutions of mineral acids – An overview

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Abstract

The review describes the current state of studies in the field of protection of ferrous and nonferrous metals from corrosion in solutions of mineral and organic acids by N-containing five-membered heterocyclic compounds (NFHC). Literature data on the protection of various metals (steels, copper-, zinc-, and aluminum-based alloys) by these corrosion inhibitors (CIs) in acid solutions are generalized. The specifics of the mechanism of their protective effect are discussed. Analysis of data on the free adsorption energy of NFHC on the surface of various metals in acid media often allows assuming the chemisorption character of their interaction, which to a great extent explains the efficiency of these compounds in corrosion inhibition. The unique ability of composite NFHC-based CIs to protect metals in extremely corrosive media (high temperature acid solutions (t up to 200°C) and acid solutions containing Fe(III) salts) is discussed. The ability of such composite CIs to inhibit corrosion in such corrosive media is based on the ability of NFHCs to form polymolecular protective layers on the metal surface, with molecules in such layers being chemically bound with each other and with the metal surface. The chemical bonds between NFHC molecules in the protective layer result from the formation of a polymeric complex compound with the metal cations formed in the course of corrosion. It is shown that it is more expedient to use not individual NFHCs but NFHC-containing mixtures. The reference list includes 260 references.

Keywords: *acid corrosion, corrosion inhibitors, metal, steel, nitrogen-containing heterocyclic compounds, pyrrole, pyrrolidine, pyrrolidone, pyrazole, pyrazoline, imidazole, imidazoline, imidazolidine, triazole, tetrazole, indole, benzimidazole, benzotriazole.*

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Introduction

In recent decades, organic heterocyclic compounds, basically represented by nitrogen-containing heterocycles, are being actively studied as corrosion inhibitors (CIs) for ferrous and nonferrous metals [1–7] in aqueous (acidic [8–13], carbonate [14, 15], hydrogen sulfide-containing [16, 17], neutral [18], alkaline [19]) and vapor-phase [20] media. Solutions of mineral and organic acids that find a wide application in various spheres of

human activity are the most important media among those characterized by high corrosivity toward metals and alloys. These spheres include metallurgy, housing and utilities infrastructure, and oil and gas complex.

Mineral acids are used to strip scale from steels. Various acid formulations are applied for efficient removal of mineral scale and rust from the inner surfaces of various process equipment (including metallic heat exchangers). To stimulate the output of raw hydrocarbons and water, oil-, gas-, and water-bearing deposits are often treated with acid solutions. In the course of these operations, an acid contacts metallic parts of the equipment used in such processes. This results in degradation of metallic parts and an increase in acid consumption. The only acceptable method for protecting metals in such media is to use CIs, *i.e.*, chemical compounds or formulations that, when present in the system in sufficient amounts, decrease the metal corrosion rate without any significant change in the concentration of any corrosive agent [21]. To date, a lot of new and important data have been published in scientific literature on the metal protection in acid media by nitrogen-containing heterocyclic compounds, which requires that these data be generalized, systematized, and analyzed in detail. This would allow one to determine the groups of such compounds that are most promising as CIs, elucidate the general regularities of the mechanism of their protective action, determine the ways of enhancing the protection efficiency, and formulate practical recommendations for the application of these CIs under real process conditions.

N-CONTAINING FIVE-COMPONENT HETEROCYCLIC COMPOUNDS

Figure 1. The most important nitrogen-containing five-membered organic heterocycles used as part of corrosion inhibitors.

Our earlier review [9] discusses in detail the options of metal protection in acid media by nitrogen-containing six-membered heterocycles. This review considers similar five-

membered structures that encompass saturated and unsaturated simple compounds (Figure 1) and complex cyclic systems including such structures [22]. Let us state from the start that we have already analyzed the specifics of metal protection in acid media by nitrogen-containing five-membered heterocyclic compounds (NFHCs) [8]. However, the interest in these compounds, the constantly expanding choice of such compounds, and important results obtained in recent years in their study made it necessary to write this paper. Review [10] analyzes the specifics of steel protection in acid media by triazole derivatives, but the data presented there mainly cover the high-temperature corrosion conditions ($t \geq 80^\circ\text{C}$).

The chemical composition of steels discussed in this review is provided in Table 1.

Table 1. Chemical composition of metals.

Metal brand	Content elements in mass percent, %
Temper-rolled low-carbon steel [23]	0.022 C; 0.004 Si; 0.14 Mn; 0.010 Cr; up to 0.002 Ni; 0.015 Cu; 0.017 S; 0.007 P; remainder Fe
Mild steel [28, 29]	0.076 C; 0.192 Mn; 0.026 Si; 0.012 P; 0.050 Cr; 0.023 Al; remainder Fe
Mild steel [31]	0.21 C; 0.05 Mn; 0.01 Si; 0.09 P; 0.05 S; 0.01 Al; remainder Fe
Mild steel [33]	0.14 C; 0.35 Mn; 0.17 Si; 0.025 S; 0.03 P; remainder Fe
Carbon steel L-52 [46]	0.26 C; 1.35 Mn; 0.05 S; 0.04 P; 0.005 Nb; 0.02 V; 0.03 Ti; remainder Fe
C38 steel [47]	0.36 C; 0.27 Si; 0.66 Mn; 0.21 Cr; 0.22 Cu; 0.02 S; 0.015 P; 0.02 Mo; remainder Fe
Mild steel [73]	0.16 C; 0.032 Mn; 0.18 Si; 0.026 S; 0.03 P; remainder Fe
Steel P110 [76]	0.26 C; 0.19 Si; 1.37 Mn; 0.004 P; 0.004 S; 0.148 Cr; 0.28 Ni; 0.019 Cu; 0.013 Mo; 0.006 V; 0.026 Al; remainder Fe
Copper [95]	0.019 Al; 0.001 Ni; 0.116 Si; 0.004 Mn; remainder Cu
Mild steel [105]	0.20 C; 0.35 Mn; 0.16 Si; 0.01 S; 0.05 P; remainder Fe
Steel 3	0.14–0.22 C; 0.15–0.33 Si; 0.40–0.65 Mn; up to 0.3 Cr; up to 0.30 Ni; up to 0.008 N; up to 0.30 Cu; up to 0.05 S; up to 0.04 P; up to 0.08 As; remainder Fe
Steel 20	0.17–0.24 C; 0.17–0.37 Si; 0.35–0.65 Mn; up to 0.25 Cr; up to 0.30 Ni; up to 0.30 Cu; up to 0.04 S; up to 0.035 P; up to 0.08 As; remainder Fe
Carbon steel [129]	1.68 C; 0.65 N; 0.10 Na; 0.07 Mg; 0.39 Cr; 0.69 Mn; remainder Fe
316 Stainless steel [139]	0.013 C; 0.3 Si; 1.4 Mn; 10.18 Ni; 0.001 S; 0.026 P; 17.2 Cr; 2.1 Mo; 0.026 N; remainder Fe
12Kh18N10T steel [140, 143]	up to 0.12 C; up to 0.8 Si; up to 2 Mn; 9–11 Ni; up to 0.02 S; up to 0.035 P; 17–19 Cr; up to 0.3 Cu; up to 0.5 Ti; remainder Fe

Metal brand	Content elements in mass percent, %
1Kh18N9T steel [141, 144]	17.57 Cr; 9.16 Ni; 71.92 Fe; 0.83 Ti; 0.52 Si; remainder Fe
08Kh18N10T steel [142]	0.08 C; 0.8 Si; up to 2 Mn; 9–11 Ni; up to 0.02 S; up to 0.035 P; 17–19 Cr; up to 0.3 Cu; up to 0.7 Ti; remainder Fe
Copper M1	Up to 0.005 Fe; up to 0.002 Ni; up to 0.004 S; up to 0.002 As; up to 0.005 Pb; up to 0.004 Zn; up to 0.05 O; up to 0.002 Sb; up to 0.001 Bi; up to 0.002 Sn; remainder Cu (99.90)
Brass L63	Up to 0.2 Fe; up to 0.001 P; up to 0.07 Pb; 34.5–38 Zn; up to 0.005 Sb; up to 0.002 Bi; remainder Cu
Zinc Ts2	Up to 0.05 Fe; up to 0.005 Cu; up to 0.01 As; up to 1 Pb; up to 0.002 Sn; up to 0.2 Cd; remainder Zn
Aluminum D16	Up to 0.5 Fe; up to 0.5 Si; 0.3–0.9 Mn; up to 0.1 Ni; up to 0.1 Ti; 3.8–4.9 Cu; 1.2–1.9 Mg; up to 0.3 Zn; remainder Al
Aluminum AMg3,5	Up to 0.4 Fe; up to 0.25 Si; up to 0.1 Mn; 0.15–0.35 Cr; up to 0.2 Ti; up to 0.1 Cu; up to 0.0008 Be; 3.1–3.9 Mg; up to 0.2 Zn; remainder Al
Mild steel Q235 [171]	0.17 C; 0.47 Mn; 0.26 Si; 0.017 S; 0.0048 P; remainder Fe
Steel N80	0.34–0.38 C; 0.20–0.35 Si; 1.45–1.7 Mn; up to 0.02 P; up to 0.015 S; up to 0.15 Cr; 0.11–0.16 V; remainder Fe
Steel (V.E.W. Boehler, Austria) [204]	0.05 C; 0.6 Mn; 0.36 P; 0.03 S; remainder Fe
Stainless steel SS304 [207]	0.044 C; 0.30 Si; 0.8 Mn; 9.60 Ni; 0.0015 S; 0.0025 P; 17.79 Cr; remainder Fe
Carbon steel [210]	0.28 C; 1.40 Mn; 0.03 S; 0.03 P; 0.06 Ti; remainder Fe
Steel 70S2KhA [232, 233]	0.7 C; 0.52 Mn; 1.52 Si; 0.3 Cr; remainder Fe

Inhibition of metal corrosion in acid media by nitrogen-containing five-membered heterocyclic compounds

Simple cyclic systems

The simplest aromatic NFHC is *pyrrole* that is characterized by high chemical reactivity. Using pyrrole as a CI of acid corrosion is of little promise since it is unstable in acid solutions: protons catalyze its polymerization to give an insoluble red resinous product [22]. Despite this, there are data on the protection of temper rolled low-carbon steel by this compound (0.1%) in 2 N H₂SO₄ (38–93°C) [23]. As it could be expected, the efficiency of the CI is low and the inhibitor's protective effect (*Z*) is 76–79%. *Z* = 95–97 and 97–99% only in the presence of 17 mM KCl and KI, respectively, though these salts provide *Z* = 4–22 and 97% by themselves.

Substituted pyrroles (2,5-difuryl-*N*-methylpyrrole [24], 1-phenyl-1*H*-pyrrole-2,5-dione, 1-(4-methylphenyl)-1*H*-pyrrole-2,5-dione [25], 1-(1-benzyl-4-(4-chlorophenyl)-2,5-dimethyl-1*H*-pyrrol-3-yl)ethanone [26]) protect different steels in cold 1 M HCl. *N*-((1*H*-pyrrol-2-yl)methylene)aniline, *N*-((1*H*-pyrrol-2-yl)methylene)-4-methylaniline, *N*-((1*H*-pyrrol-2-yl)methylene)-4-methoxyaniline, and *N*-((1*H*-pyrrol-2-yl)methylene)-3,5-dimethylaniline at a content of 500–2000 ppm in hot 10% HCl (60°C) considerably inhibit mild steel corrosion, providing $Z = 95.8\text{--}99.6\%$ [27]. In 1 M HCl (35°C), 2-amino-3-methyl-3-phenyl-5-(phenylthio)-3*H*-pyrrole-4-carbonitrile, 2-amino-3-(4-hydroxyphenyl)-3-methyl-5-(phenylthio)-3*H*-pyrrole-4-carbonitrile, 2-amino-3-(2,4-dihydroxyphenyl)-3-methyl-5-(phenylthio)-3*H*-pyrrole-4-carbonitrile [28], and 2-amino-3-methyl-3-(4-nitrophenyl)-5-(phenylthio)-3*H*-pyrrole-4-carbonitrile [29] at a content of 50 mg/L slow down the corrosion of mild steel considerably ($Z = 94.4\text{--}98.7\%$), but their protective effect decreases with an increase in temperature (t).

Low-molecular *pyrrolidine* derivatives weakly suppress iron corrosion in 6.1 M HCl (30°C). According to voltammetric data, the protective effect of CIs grows in a series of *N*-methylpyrrolidine < *N*-ethylpyrrolidine < pyrrolidine. Besides, $Z = 60\%$ in the presence of 10 mM pyrrolidine in a solution [30]. In 0.5 M H₂SO₄ (25°C), 1-*R*-pyrrolidine-2-one ($R = -\text{CH}=\text{CH}_2$, $-(\text{CH}_2)_2-\text{S}-(\text{CH}_2)_2-\text{OH}$, $-(\text{CH}_2)_2-\text{S}-\text{CH}_2-\text{COOH}$ [31], $-(\text{CH}_2)_2-\text{P}(\text{O})(\text{OC}_2\text{H}_5)_2$ [32]) also weakly inhibits steel corrosion ($Z_{\text{max}} = 71\text{--}89\%$). Despite the presence of a bulky substituent in the pyrrolidine ring in 1-((pyridin-2-ylamino)(pyridin-4-yl)methyl)-pyrrolidine-2,5-dione, it is also inefficient in the inhibition of mild steel corrosion in 1 M HCl [33]. Only quaternized salts (*N,N*-dipropargylpyrrolidinium bromide, *N*-dodecyl-*N*-propargylpyrrolidinium bromide, and *N*-hexadecyl-*N*-propargylpyrrolidinium bromide (25–50 ppm)) can efficiently protect low-carbon steel in 1 M HCl at 25°C ($Z \geq 95\%$) [34]. The observed effect is largely determined by the presence in these compounds of a propargyl moiety that is very reactive and can cause polymerization of CI molecules on the metal surface to form a protective polymer layer [8, 35–37]. Ammonium and sodium pyrrolidine dithiocarboxylates that provide Z up to 90% were studied in 0.5 M HCl and 5% HCl as CIs for copper and Monel (composition, wt.%: 70 Ni and 30 Cu) [38, 39]. Vinylpyrrolidone-based polymer and copolymer additives are used for the protection of mild and carbon steels in HCl and H₂SO₄ solutions [40–45], but even though these compounds provide efficient metal protection in cold acid solutions, they fail to do so upon temperature increase.

The scope of five-membered nitrogen-containing CIs with two heteroatoms is more extensive. Imidazole itself weakly suppresses steel corrosion [46–48]. According to voltammetric data, the addition of 5 mM imidazole provides only $Z = 83\%$ for carbon steel L-52 in 0.5 M H₂SO₄ (30°C) [46]. In 1 M HCl in the presence of 4% imidazole, $Z = 60\%$ for steel C38 [47]. Similarly, imidazoles with small substituents fail to provide good protection of steels in HCl solutions [49–51]. In contrast, imidazoles with four phenyl substituents:

provide efficient corrosion inhibition of low-carbon steel in 1 M HCl and 0.5 M H₂SO₄ [52, 53]. In recent years, imidazole derivatives with quaternized nitrogen atoms in the heterocycle – Gemini surfactants [54], imidazole zwitterions [55–57], and ionic liquids [58–67] have been actively used as steel CIs in acids. Imidazole derivatives are considered as CIs for nonferrous metals: copper in H₂SO₄ [68, 69], HCl [70] and HNO₃ [71], aluminum in HCl [72].

As opposed to compounds containing an aromatic imidazole ring, imidazoline [73–79] and *imidazolidine* [80] derivatives are less studied as CIs. Specifically, 3,4-dimethoxyphenyl-2-imidazoline (40 ppm) on low-carbon steel in 0.5 M H₂SO₄ (30°C) provides $Z=58.3\%$ according to the data of three-hour tests. The best result is observed with 4-methoxyvinyl-2-imidazoline, where $Z=82.4\%$ at a lower content in the corrosion medium (20 ppm) [73]. Addition of 0.4 g/L 2-heptadecyl-1-[2-(octadecanoylamino)ethyl]-2-imidazoline poorly protects steel St37 (Erdemir Steel Co., Turkey) in 15% HCl (25°C), as shown by electrochemical impedance spectroscopy (EIS) data: $Z=47.9\%$ [74]. Under similar conditions, $Z=89.9\%$ for *N*-(2-(2-tridecyl-4,5-dihydro-1*H*-imidazol-1-yl)ethyl)tetradecanamide [75]. Of interest is 2-methyl-4-phenyl-1-tosyl-4,5-dihydro-1*H*-imidazole that provides $Z=94.6\%$ at a content of 0.3 g/L in 1 M HCl (60°C) according to the data of 72-hour tests on carbon steel P110 [76]. A high-molecular imidazolidine derivative, 3-methyl-5,5'-diphenylimidazolidine-2,4-dione, poorly inhibits mild steel corrosion according to the data of gravimetric studies in 1 M HCl (25°C) [80].

Pyrazole that is an aromatic system is an inefficient CI. According to voltammetric data, iron (99.995%) corrosion in 1 M HCl (25°C) containing 10 mM pyrazole is inhibited only 3.4-fold [81]. Using pyrazole derivatives with various substituents, good steel protection can be provided, predominantly in HCl solutions [82–92] and, more rarely, in H₂SO₄ solutions [93]. Pyrazole derivatives can be used for the protection of nonferrous metals in acid media. 5-Amino-3-[4'-chlorophenyl]pyrazole and 5-amino-3-[4'-methylphenyl]pyrazole in 0.06 M HCl (25°C) provide $Z=89\%$ for zinc [94]. Copper corrosion in 2 M HNO₃ (25°C) is inhibited almost 10-fold in the presence of $11 \cdot 10^{-6}$ M 3'-(3,4-dichlorophenyl)-2',3',6,7,8,9-hexahydro-2'-phenyl-5'-styrylspiro[benzocyclo-heptene-6(5*H*),4'(4*H*-pyrazol)]-5-one, but an increase in the corrosion medium temperature weakens the protective effects [95]. Derivatives of a more saturated pyrazoline ring were also studied as steel CIs in acids [96–100].

Active studies of *triazoles* as CIs of metals in acid media were covered in recent review papers [101, 102] that are largely a collection of reference data. Substituted 1,2,3-triazoles

[103–109] and 4,5-dihydro-1*H*-1,2,3-triazole [110] are predominantly used for the protection of low-carbon and carbon steels in HCl solutions. Of interest is (2*R*)-benzyl-2-amino-3-(((1-(((2*R*,3*R*,4*S*,5*S*,6*S*)-3,4,5-*tris*(benzyloxy)-6-methoxytetrahydro-2*H*-pyran-2-yl)methyl)-1*H*-1,2,3-triazol-4-yl)methoxy) butanoate that provides $Z=97.0\text{--}99.4\%$ in 2–6 M HCl at 25°C [105].

1,2,4-Triazoles are studied better. 3,5-*Bis*(*R*)-4-amino-1,2,4-triazole (*R*=4-methoxyphenyl [111], *m*-tolyl [112], methoxy [113], 2-thienylmethyl [114], pyridyl [115], 4-chlorophenyl [116], or 3,4-dimethoxyphenyl [117]) are used to protect mild steel in HCl and H₃PO₄ solutions. Azomethines with various structures synthesized on the basis of 4-amino-1,2,4-triazole [118–122] and 3-amino-1,2,4-triazole derivatives [123] are mild steel corrosion inhibitors in HCl solutions. Earlier [124], we pointed out the high efficiency of azomethines containing moieties of cinnamic aldehyde and a nitrogen-containing heterocycle in the inhibition of steel corrosion in hot ($t \leq 105^\circ\text{C}$) HCl solutions. Indeed, in 2 M HCl (20–100°C), the addition of 5 mM *N*-cinnamylidene-1*H*-1,2,4-triazole-3-amine suppresses the corrosion of steel 3 by a factor of 5.1–278 [125]. With an increase in *t*, the protective effect of this compound is only enhanced, which allows one to consider it as a high-temperature CI [36, 126]. Under similar conditions, the additive of the well-known CI, cinnamic aldehyde [127] used in the synthesis of this compound, inhibits corrosion only 4.2–24 fold. Moreover, the temperature maximum of its efficiency is at 90°C, which is a poorer result [125]. Parallel studies of 3-amino-1,2,4-triazole showed its weak inhibitive properties ($\gamma \leq 2.3$).

1,2,4-Triazole derivatives with bulky substituents are characterized by high efficiency in steel protection in HCl solutions. A Gemini surfactant, 3,5-*bis*(methylene octadecyl dimethylammonium chloride)-1,2,4-triazole (1 mM), in 1 M HCl provides $Z=98.3\%$ for carbon steel at 25°C [128]. High-molecular compounds based on 1,2,4-triazole often contain sulfur [129, 130]. In the presence of 1 mM 5-hexylsulfanyl-1,2,4-triazole, 12 h exposure of a carbon steel sample in this medium provides $Z=93\%$ [129].

The capabilities of 1,2,4-triazole derivatives in preventing steel corrosion in acid media are unique to a large extent. In fact, 3-substituted 1,2,4-triazole derivatives with various structures (inhibitors such as IFKhAN-92 [131–133], IFKhAN-93 [134], IFKhAN-94 [135], as well as IFKhAN-96 that is a 4-substituted 1,2,4-triazole [136]) inhibit the corrosion of mild steels in HCl and H₂SO₄ solutions at *t* up to 100°C in many cases [10]. They demonstrate a protective effect in a wide range of acid concentrations, $C=1\text{--}8\text{ M}$ [134–136]. At $t=60^\circ\text{C}$, the addition of 5 mM IFKhAN-94 inhibits the corrosion of steel 3 by a factor of 24.8–121 in 1–8 M HCl and 32.9–905 in 1–8 M H₂SO₄ [135]. It is important for practical purposes that while triazole derivatives efficiently inhibit steel corrosion, they do not slow down the removal of thermal scale in acids [137]. It has been shown for CIs IFKhAN-92 and IFKhAN-93 as examples [138] that triazole derivatives efficiently suppress steel hydrogenation in addition to considerable inhibition of its corrosion in HCl and H₂SO₄ solutions.

Chromium-nickel stainless steels are unstable in (especially hot) HCl and H₂SO₄ solutions. There are problems regarding their protection by inhibitors in these media,

including the protection by individual triazoles. In fact, at 2000 ppm, 4-{{[4-(dimethylamino)benzylidene]amino}-5-methyl-4*H*-1,2,4-triazole-3-thiol} in 2 M HCl (30–50°C) provides $Z=69.8\text{--}91.8\%$ on stainless steel 316 according to voltammetric data [139]. Of more interest is IFKhAN-92 that considerably inhibits the corrosion and hydrogenation of chromium-nickel steels (12Kh18N10T, 1Kh18N9T, 08Kh18N10T) at $C=5\text{ mM}$ in hot HCl solutions, including those containing H_2S [140–142]. Steel protection by IFKhAN-92 in H_2SO_4 solutions is considerably enhanced in the presence of H_2S [143, 144].

1,2,4-Triazole derivatives protect not only ferrous but also nonferrous metals in acid media. In HCl solutions, the addition of 4-amino-5-alkyl-3-mercapto-1,2,4-triazoles (alkyl = methyl, ethyl, propyl) inhibits the corrosion of copper (99.9%) [145] and Muntz metal (60% Cu, 40% Zn) [146]. 3,5-Bis(4-methylphenyl)-4-amino-1,2,4-triazole and 3,5-bis(4-methoxyphenyl)-4-amino-1,2,4-triazole protect copper (99.9%) in 1 M HCl (30°C) [147]. IFKhAN-92 protects a wide range of nonferrous metals in mineral acids [148]. For copper M1 in 2 M HCl (room temperature) at $C_{\text{in}}=5\text{ mM}$ and exposure duration of 7 days, $Z=97.7\%$. In 2 M H_2SO_4 and HCl (95°C) for brass L63 at the same C_{in} , $Z=78$ and 86%. Here, the inhibitor prevents the dezincification of the metal surface that is observed in the background media. In 2 M H_2SO_4 , HCl, and H_3PO_4 (25°C), the corrosion rates (k) of zinc Ts2 are 753, 1429, and 121 g/(m²·h), respectively, against only 3.1, 3.0, and 4.6 g/(m²·h) in the presence of 5 mM IFKhAN-92. In 2 M HCl (25°C), the k of aluminum alloys D16 and AMg3.5 are 1425 and 97.2 g/(m²·h) against 3.0 and 0.7 g/(m²·h) in the presence of 10 mM IFKhAN-92.

In 1 M HCl (25°C), *tetrazole* (2 mM) stimulates the corrosion of pure iron (99.96%) by accelerating the anodic and especially cathodic reactions on the metal, but its substituted derivative, 1-phenyl-5-mercapto-1,2,3,4-tetrazole, provides $Z=98.2\%$ [149]. Substituted tetrazoles also suppress steel corrosion in HCl solutions [150–153]. Tetrazole derivatives (organic cations [154–159] and thiols [160, 161]) were studied in H_2SO_4 solutions as steel CIs. Tetrazoles can protect steels in a wide temperature range. Addition of 5 mM IFKhAN-95 (a 5-substituted tetrazole) in 2 M H_2SO_4 and HCl (25–95°C) inhibits the corrosion of steel 3 by a factor of 35–149 and 19–111, respectively. The maximum CI efficiency for H_2SO_4 is at 80°C and is not achieved for HCl under experimental conditions, which is characteristic of high-temperature CIs [162]. Organic tetrazole-based cations inhibit steel corrosion in acetic acid solutions [163] and in dilute solutions of chloroacetic acids (ClCH_2COOH , Cl_2CHCOOH , and Cl_3CCOOH) [164]. Tetrazole-containing compounds are inhibitors of copper corrosion in solutions of HCl [165, 166], H_2SO_4 [167, 168], and HNO_3 [169], and of aluminum corrosion in 1 M HCl [170].

Complex systems

The simplest fused system containing a pyrrole moiety is *indole* that significantly inhibits the corrosion of mild steel Q235 in 0.1 M H_2SO_4 ($Z=93.7\text{--}97.2\%$) at $C_{\text{in}}=4\text{--}8\text{ mM}$ [171]. In 1 M H_2SO_4 , 5-aminoindole and 5-chloroindole provide weaker corrosion inhibition of steel than indole [172]. The efficiency of the protection of pure iron (Puratronic, 99.9999%)

in 1 M HCl (25°C) by nitrogen-containing heterocycles decreases in the series indole > benzotriazole > benzothiazole > benzoimidazole [173]. The corrosion of mild steels in acids is inhibited by indole-containing amines [174], azomethines [175], and ionic liquids [176]. Indole carboxylic acids were studied as CIs for mild steel in solutions of HCl [177] and H₂SO₄ [178] and for copper in 0.5 M H₂SO₄ [179]. More complex substituted cyclic systems, such as 3,6-dibromo-9-phenylcarbazole, 3(9*H*-carbazol-9-yl)-1,2-propanediol [180], 2,3,4,9-tetrahydro-1*H*-carbazole-8-carboxylic acid, 6-methyl-2,3,4,9-tetrahydro-1*H*-carbazole, 9-methyl-9*H*-carbazole-3-carboxylic acid [181], 9*H*-pyrido[3,4-*b*]indole, 1-methyl-9*H*-pyrido[3,4-*b*]indole [182], and 1,4-*bis*(2-pyridyl)-5*H*-pyridazino[4,5-*b*]indole [183] were studied as mild steel CIs in cold 1 M HCl. $Z > 90\%$ was observed in the C_{in} range studied only with the latter compound. Two indolizine derivatives are of interest:

They protect steel N80 in 15–20% HCl and 12% HCl + 3% HF (90°C) where the background steel corrosion rates are $k_0 = 1243\text{--}1648$ and $1105\text{ g}/(\text{m}^2\cdot\text{h})$, respectively [184]. In the presence of 1% compound (I), $k = 2.0\text{--}10.6$ and $2.1\text{ g}/(\text{m}^2\cdot\text{h})$, while in the presence of compound (II), $k = 3.2\text{--}12.2$ and $2.1\text{ g}/(\text{m}^2\cdot\text{h})$. With a t increase to 120°C, the protective effect of these compounds deteriorates, especially in 20% HCl [185].

Individual *benzimidazole* (BI) manifests a weak protective effect in low-carbon steel protection in 1 M HCl (20°C). 2-Substituted BIs are more efficient. At $C_{in} = 1\text{ mM}$, steel corrosion inhibition is enhanced in the series BI < 2-methyl-BI < 2-hydroxymethyl-BI < 2-amino-BI < 2-acetonitrile-BI < 2-mercapto-BI. For the most efficient CIs, namely 2-amino-BI, 2-acetonitrile-BI, and 2-mercapto-BI, the maximum degrees of protection observed in hydrochloric acid are $Z_{max} = 94.0, 94.0,$ and 94.7% [186]. However, the Z of even for the best of these CIs, 2-mercapto-BI (1 mM), decreases from 97 to 88% for mild steel with an increase in t from 40 to 80°C in the same medium [187]. In many cases, BIs with bulky substituents of various nature [188–196] and ionic liquids on its basis [197, 198] are promising for steel protection in HCl solutions. Quaternized BIs protect mild steel in 0.5 M H₂SO₄ (25–55°C) [199] and a 6061 Al – 15 vol.% SiC_(p) composite in 0.1 M HCl and H₂SO₄ (30–50°C) [200]. Substituted derivatives of *imidazo[1,2-*a*]pyridine*

inhibit steel corrosion in a wide range of mineral acids (HCl, H₂SO₄, H₃PO₄) [6, 201].

Derivatives of pyrazolo-pyrimidine

were studied as corrosion inhibitors for pure copper in 0.5 M H_2SO_4 [202, 203]. The most efficient one, 4-mercapto-pyrazolo-pyrimidine, provides $Z=91\%$ at $C_{\text{in}}=0.5$ mM [203].

1,2,3-Benzotriazole (BTA) widely used as a CI [2–4] was also studied in acid media [204, 205]. However, even in dilute 0.1 M H_2SO_4 (30°C), 0.01–0.09 M BTA weakly inhibits mild steel corrosion, $Z=76.9$ –89.2% [204]. In the presence of 0.01 M BTA, an increase in t from 30 to 65°C causes a decrease in Z from 76.9 to 33.5%. Addition of CuSO_4 that enhances the protective effect of BTA can provide high $Z=99.4\%$ (0.01 M BTA with 0.001 M CuSO_4) at $t=30^\circ\text{C}$. A mixture of BTA with Cu(II) is more efficient in iron protection in 0.5 M H_2SO_4 (30°C) [205]. The practical application of CI mixtures containing Cu(II) salts for steel protection can be dangerous. A reaction of Cu(II) cations with the metal results in the formation of metallic copper on its surface and appearance of Cu–steel galvanic couples. This may result in corrosion localization in acid media and may adversely affect the further operation of steel structures. A mixture of BTA with disodium ethylenediamine tetraacetate is used for the protection of cold rolled steel in dilute 0.1 M HCl (30°C) [206].

According to voltammetric data, BTA (10 mM) protects stainless steel SS304 in 0.1 M H_2SO_4 (30–70°C), providing $Z=86.7$ –99.1%. Importantly, an increase in t results in a systematic increase in Z [207]. Unsatisfactory results were obtained in the protection of low carbon steel in cold 1 M HCl and 1 M HCl + 3.5% NaCl by 100 ppm 5-methyl-1*H*-benzotriazole, whose Z does not reach 90% even in combination with KI, a well known synergetic additive (5 mM). BTA derivatives with bulkier substituents are efficient in corrosion inhibition of Armco iron and mild steel in H_2SO_4 solutions [208, 209]. An ionic liquid, 1-dodecyl-5-methyl-1*H*-benzo[d][1,2,3]triazol-1-ium bromide, strongly inhibits carbon steel corrosion in cold 7 M H_3PO_4 . However, for 1 mM of this compound, an increase in t from 20 to 80°C causes a decrease in Z from 97.2 to 87.8% [210]. BTA and its derivatives were studied as corrosion inhibitors of copper and its alloys in HCl, H_2SO_4 , and HNO_3 solutions [211–217]. Among the compounds studied, 1-(phenylsulfonyl)-1*H*-benzotriazole [211] and benzotriazole-1-carboxamide [212] are of interest as that provide $Z=93.5$ and 92.4% for copper, respectively, even at relatively low concentrations (5 mM) in corrosive 1 M HNO_3 (25°C). In 0.5 M HCl (30°C), copper corrosion is inhibited 27.5-fold at a high content (50 mM) of *N*-(2-thiazolyl)-1*H*-benzotriazole-1-carbothioamide [213].

The compound with a complex structure

is a weak CI for low-carbon steel in 0.5 M H_3PO_4 that provides only $Z = 71.5\%$ at $C_{\text{in}} = 0.1 \text{ M}$ [218].

Thus, a rather wide range of NFHCs including both simple and more complex systems have been studied as CIs of metals in acid media. These compounds are mainly recommended for the corrosion inhibition of various steels, though it is pointed out that they can be used for protection of nonferrous metals, chiefly copper and copper-based alloys. The effect of these CIs is manifested in solutions of HCl , H_2SO_4 , H_3PO_4 , and, in some cases, even HNO_3 . Unfortunately, no systematic studies on the effect of the structure of NFHCs on their protection from metal corrosion in acid media are provided in scientific literature. However, even the available data allow us to draw the conclusion that the efficiency of the heterocyclic systems under consideration increases when bulky substituents are introduced into their structure. However, this is not always the case.

It is worthy of note that many of the compounds considered as CIs in this review are difficult to obtain and expensive, which considerably limits the prospects of their industrial application. Besides, industrial acid treatment conditions imply that they should be used at elevated temperatures. As the data on metal protection by CIs provided here either correspond to cold solutions or demonstrate a decrease in protection with an increase in t , only some of the compounds are of particular interest. For example, CIs of the IFKhAN series can enhance the protective effect in steel corrosion up to temperatures close to 100°C , which is promising for their industrial use. The efficiency of the CIs under consideration can be enhanced by chemical modification of their molecular structure, which significantly increases their cost. This way is of little promise. An alternative is to develop mixtures with other compounds based on the already studied heterocyclic CIs, which does not lead to any considerable rise in their cost, expands the range of their application, and decreases their consumption. Certain important results in this direction have already been achieved, as described in the next section.

Composite CIs based on nitrogen-containing five-membered heterocyclic compounds

This section considers the most outstanding results in metal protection in acid media by composite NFHC-based CIs. A conventional method of enhancing the protective effect of organic CIs is to develop binary formulations with metal halides on their basis [219]. Indeed, binary formulations such as IFKhAN-92 + KI and IFKhAN-92 + KBr can protect steel 20 in H_2SO_4 solutions at $t \leq 200^\circ\text{C}$ [133, 220]. The application of the IFKhAN-92 + urotropine mixture in HCl solutions allows one to significantly inhibit the corrosion of steel 20 at $t \leq 160^\circ\text{C}$ [132, 220]. A more interesting result is provided by a three-component mixture of IFKhAN-92 + KI + urotropine that protects not only low-carbon, but also, most notably, chromium-nickel steels [142, 221, 222] under high-temperature acid corrosion conditions ($t > 100^\circ\text{C}$). Importantly, it is sufficient to use binary formulations of IFKhAN-92 + KI, IFKhAN-92 + KBr (H_2SO_4 solutions), and IFKhAN-92 + urotropine (HCl solutions) in acid solutions with $t \leq 100^\circ\text{C}$ for the protection of chromium-nickel steels [140, 141, 143, 144].

More detailed information on steel protection in HCl and H₂SO₄ solutions ($t = 0\text{--}200^\circ\text{C}$) by composite triazole-based CIs is summarized in review [10].

The next group of composite NFHC-containing CIs are binary mixtures of sulfur-containing molecular (thiourea, phenylthiourea, *N*-tolylthiourea, diphenylthiourea, Captax) and anionic (sodium diethyldithiocarbamate (SDEDTC), KNCS) additives. They enhance the protective effect of substituted triazoles on steel corrosion in H₂SO₄ solutions [125, 134, 135, 223]. For example, the addition of 10 mM *N*-cinnamylidene-1*H*-1,2,4-triazole-3-amine (CTA) inhibits corrosion of steel 3 in 2 M H₂SO₄ (60°C) only 2.2-fold, while the 5 mM CTA + 5 mM SDEDTC and 5 mM CTA + 5 mM KNCS formulations provide 83- and 65-fold inhibition. Individual 10 mM SDEDTC and 10 mM KNCS decrease the corrosion rate only 1.1- and 2.6-fold under similar conditions [125]. In solutions of phosphoric, formic, acetic, and citric acids, substituted triazoles weakly inhibit the corrosion of low-carbon steels. However, a substituted triazole, IFKhAN-92, and sulfur-containing additives use simultaneously allow one to protect steels in these media efficiently. Of particular interest is the formulation of IFKhAN-92 and KNCS that protects low-carbon steels in H₃PO₄ solutions [224] and citric acid [225] at temperatures up to 100°C. Under similar conditions, the IFKhAN-92+SDEDTC formulation inhibits low-carbon steel corrosion in formic and acetic acid solutions [226].

The use of HCl and H₂SO₄ solutions leads to accumulation of Fe(III) salts in these systems due to dissolution of rust or thermal scale in acids. Steel protection in solutions of such acids containing Fe(III) salts poses a serious problem. In these media, CIs inhibit the reduction of protons on steels, but fail to inhibit the reduction of such a strong oxidant as Fe(III), which renders the inhibitory protection of the metal unsatisfactory [227]. A unique property of the formulation of IFKhAN-92+KNCS is its ability to slow down the corrosion of low-carbon steel in H₃PO₄ solutions containing FePO₄ [228]. It turns out that efficient steel protection ($t = 20\text{--}80^\circ\text{C}$) by the formulation of IFKhAN-92 + KNCS can be provided not only in H₃PO₄ solutions but also in H₂SO₄ + H₃PO₄ + Fe(III) mixtures containing as little as 40% H₃PO₄ [229, 230]. A more interesting result is provided by a three-component CI comprising IFKhAN-92 + KNCS + urotropine that also inhibits steel corrosion in HCl + H₃PO₄ + Fe(III) mixtures containing 40% H₃PO₄ [230, 231]. A similar effect of the formulation of IFKhAN-92 + KNCS + urotropine is observed for high-strength steel 70S2KhA in H₂SO₄ + H₃PO₄ + Fe(III) [232] and in HCl + H₃PO₄ + Fe(III) [233]. It is known that 70S2KhA steel shows a tendency for hydrogen absorption in acid media. It was shown that the IFKhAN-92-based three-component formulation considerably suppresses hydrogen absorption by 70S2KhA steel. These results are very important for practical purposes since they open new ways for the development of inhibited acid solutions with a wide temperature range that are stable against accumulation of Fe(III) cations and are based on relatively cheap HCl and H₂SO₄.

Thus, the possibility of developing new high-performance CIs was shown for formulations based on triazole derivatives in a wide range of acid media where they can be used. Composite CIs open new prospects for practical application of NFHCs in the fields

where they were never used before: high-temperature steel CIs in acid media and CIs of steel in acid solutions containing Fe(III) salts.

Regularities of the mechanism of action of nitrogen-containing five-membered heterocyclic CIs of metals in acid media

Efficient protection of metals by NFHCs in highly corrosive acid solutions, particularly under high-temperature corrosion conditions or in the presence of corrosion promoters (Fe(III) salts), is possible only if the CIs form polymolecular protective layers on the metal surface strongly bound to the latter. A strong bond between a protective CI layer and a metal surface is provided by chemisorption interaction between a CI monolayer and the metal. The molecules in a CI layer can be bound both physically and chemically. The strongest protective layers are formed by chemically bound CI molecules. There are two ways in which such protective layers are formed: by polymerization of molecules of unsaturated organic compounds [8, 35, 36] and by formation of polymeric complex compounds from CI molecules and metal cations formed due to corrosion [10]. The second way is possible if the CIs used are NFHCs that are able to form polymeric complexes with cations of *d*-metals [234–239]. Importantly, nitrogen-containing heterocycles in such complex compounds are bidentate ligands. As CIs under consideration manifest the strongest protective effects when used in combinations with other compounds, it is necessary to understand the effect of these substances on the corrosion process.

Figure 2. Dependence of coefficient of residual protective effect (γ) on time (τ) at different concentrations of inhibitor, mM: 1 – 10.0; 2 – 5.0; 3 – 2.5; 4 – 1.25; 5 – 0.625 [240].

An experimental proof that NFHCs form protective layers on the metal surface in acid solutions is given by the protective aftereffect of these CIs. It was shown [240] that iron samples exposed to 1 M H_2SO_4 (room temperature, 24 h) inhibited by 1-methyl-3-(3-phthalimido-2-hydroxypropyl)-2-iminobenzimidazoline, after washing in distilled water and transferring them to pure 1 M H_2SO_4 , corroded at a smaller rate than that of samples not conditioned in the inhibited acid (Figure 2). The protective aftereffect of this CI linearly

decreases with time and lasts from 23 to 38 h. It is the stronger the higher the C_{in} in the preadsorption solution.

The formation of a polymolecular CI layer on a metal that can provide its efficient protection begins with the formation of a CI monolayer that is chemically bound to the metal surface. The nature of the bond between a CI and a metal can be understood on the basis of its free adsorption energy ($-\Delta G_{ads}$). It is assumed that if $(-\Delta G_{ads}) < 20$ kJ/mol, its is of physical nature. Only if $(-\Delta G_{ads}) \geq 40$ kJ/mol, one can state the high probability that the CI is chemisorbed.

To understand the nature of NFHC interaction with the surfaces of metals in acid solutions, one should systematize the data on their $(-\Delta G_{ads})$. Despite the practically complete absence of any estimates of NFHC adsorption on metals in acid solutions obtained by direct measurements in the available literature, there is a lot of data on the determination of $(-\Delta G_{ads})$ obtained by a less valid method, namely, processing of the results of measurements of metal sample mass losses in inhibited acid solutions. Our analysis of literature data (Table 2) shows that the calculated $(-\Delta G_{ads})$ values for some NFHCs reach or exceed the threshold value of 40 kJ/mol, which allows assuming their chemisorption on metals.

Table 2. Free adsorption energies of nitrogen-containing five-membered organic heterocycles on metals from acid media.

Inhibitor	Type of the metal	Type of the corrosive medium	Standard adsorption free energy ($-\Delta G_{ads}$)*, kJ/mol	Ref.
Pyrroles				
2,5-Difuryl-N-methylpyrrole	Mild steel	1 M HCl (35°C)	40.8	[24]
1-(4-Methylphenyl)-1 <i>H</i> -pyrrole-2,5-dione; 1-phenyl-1 <i>H</i> -pyrrole-2,5-dione	Carbon steel	1 M HCl (room <i>t</i>)	43.9; 45.2	[25]
2-Amino-3-methyl-3-phenyl-5-(phenylthio)-3 <i>H</i> -pyrrole-4-carbonitrile; 2-amino-3-(4-hydroxyphenyl)-3-methyl-5-(phenylthio)-3 <i>H</i> -pyrrole-4-carbonitrile; 2-amino-3-(2,4-dihydroxyphenyl)-3-methyl-5-(phenylthio)-3 <i>H</i> -pyrrole-4-carbonitrile	Mild steel	1 M HCl (35–65°C)	33.8–35.7; 34.4–37.3; 35.4–39.6	[28]
2-Amino-3-methyl-3-(4-nitrophenyl)-5-(phenylthio)-3 <i>H</i> -pyrrole-4-carbonitrile	Mild steel	1 M HCl (35–65°C)	34.3–39.5	[29]

Inhibitor	Type of the metal	Type of the corrosive medium	Standard adsorption free energy ($-\Delta G_{\text{ads}}^*$), kJ/mol	Ref.
Pyrrolidines				
Diethyl-2-(2-oxopyrrolidin-1-yl)ethylphosphonate	Mild steel	0.5 M H ₂ SO ₄ (25°C)	29.9	[32]
1-((Pyridin-2-ylamino)(pyridin-4-yl)methyl)pyrrolidine-2,5-dione	Mild steel	1 M HCl (32–62°C)	32.9–36.1	[33]
<i>N,N</i> -Dipropargylpyrrolidinium bromide; <i>N</i> -dodecyl- <i>N</i> -propargylpyrrolidinium bromide; <i>N</i> -hexadecyl- <i>N</i> -propargylpyrrolidinium bromide	Mild steel	1 M HCl (25°C)	39.4; 42.1; 43.6	[34]
Sodium pyrrolidine dithiocarboxylate	Monel copper alloy	5% HCl (room <i>t</i>)	31	[39]
Polyvinyl pyrrolidone	Mild steel	1 M H ₂ SO ₄ (30–60°C)	11.2–13.1 (Temkin adsorption isotherm) 10.3–16.4 (El-Awady adsorption isotherm)	[40]
Polyvinyl pyrrolidone	Carbon steel	1 M HCl (30°C)	31.6	[41]
Co-poly(maleic anhydride–N-vinyl-2-pyrrolidone)	Mild steel	2 M H ₂ SO ₄ (25–50°C)	30.7–31.7	[42]
Polyvinyl pyrrolidone	Mild steel	0.5 M H ₂ SO ₄ (30–60°C)	4.5–12.3 (Langmuir adsorption isotherm) 17.4–18.6 (Temkin adsorption isotherm)	[43]
Poly-(vinylpyrrolidone–methylaniline)	Mild steel	1 M HCl (30–70°C)	16.7–22.2 (El-Awady adsorption isotherm)	[44]

Inhibitor	Type of the metal	Type of the corrosive medium	Standard adsorption free energy ($-\Delta G_{\text{ads}}^*$), kJ/mol	Ref.
Imidazoles				
2-Ethyl-4-methylimidazole; 1-benzylimidazole	API X65 steel	1 M HCl (room <i>t</i>)	26.1	[49]
2-(1,4,5-Triphenyl-1 <i>H</i> -imidazol-2-yl)phenol; 3-methoxy-4-(1,4,5-triphenyl-1 <i>H</i> -imidazol-2-yl)phenol	Mild steel	1 M HCl (25°C)	45.8; 45.4	[52]
2-(4-Chlorophenyl)-1,4,5-triphenyl-1 <i>H</i> -imidazole; 1,4,5-triphenyl-2-(<i>p</i> -tolyl)-1 <i>H</i> -imidazole	Mild steel	0.5 M H ₂ SO ₄ (25°C)	42.1; 41.7	[53]
Imidazolium Gemini surfactant [C ₁₄ -4-C ₁₄ im]Br ₂ ; imidazolium surfactant [C ₁₄ mim]Br	A3 carbon steel	HCl (25–55°C)	47.2–58.4; 41.3–44.0	[54]
2-(3-(Carboxymethyl)-1 <i>H</i> -imidazol-3-ium-1-yl)acetate; 2-(3-(1-carboxyethyl)-1 <i>H</i> -imidazol-3-ium-1-yl)propanoate; 2-(3-(1-carboxy-2-phenylethyl)-1 <i>H</i> -imidazol-3-ium-1-yl)-3-phenylpropanoate	Mild steel	1 M HCl (35°C)	37.0; 37.5; 37.6	[55]
2-(3-(1-Caboxy-2-hydroxyethyl)-1 <i>H</i> -imidazol-3-ium-1-yl)-3-hydroxypropanoate; 2-(3-(1-caboxy-3-methylthio)propyl)-1 <i>H</i> -imidazol-3-ium-1-yl)-4-mercaptobutanoate	Mild steel	1 M HCl (30°C)	37.6; 41.4	[57]
1,3-Dioctadecylimidazolium bromide	Mild steel	1 M H ₂ SO ₄ (room <i>t</i>)	37.9	[58]
1-Ethyl-3-methylimidazolium tetrafluoroborate; 1-butyl-2,3-dimethylimidazolium tetrafluoroborate; 1-decyl-3-methylimidazolium tetrafluoroborate	Mild steel	1 M HCl	33.7; 34.2; 36.6	[59]
1,4-Di[1-methylene-3-methylimidazolium bromide]benzene	Mild steel	1 M H ₂ SO ₄ (22°C)	17.5	[60]
1-Vinyl-3-aminopropylimidazolium hexafluorophosphate; 1-vinyl-3-aminopropylimidazolium tetrafluoroborate	Carbon steel	1 M HCl (25–45°C)	33.0–34.9; 29.9–30.4	[61]

Inhibitor	Type of the metal	Type of the corrosive medium	Standard adsorption free energy ($-\Delta G_{\text{ads}}^*$), kJ/mol	Ref.
1-Vinyl-3-methylimidazolium iodide; 1-vinyl-3-propylimidazolium iodide; 1-vinyl-3-butyylimidazolium iodide	X70 steel	0.5 M H ₂ SO ₄ (25–45°C)	30.9–31.0; 31.3–31.4; 31.1–31.9	[63]
1-Butyl-3-methylimidazolium chloride; 1-butyl-3-methylimidazolium acetate; 1-butyl-3-methylimidazolium trifluoromethanesulphonate	Mild steel	1 M HCl	33.1–34.9; 32.9–36.2; 34.0–37.0	[64]
Poly[3-butyl-1-vinylimidazolium bromide	Mild steel	1 M HCl (25°C)	25.1 (Frumkin adsorption isotherm)	[65]
3-Hexadecyl-1-methyl-1 <i>H</i> -imidazol-3- ium bromide; 3-hexadecyl-1,2- dimethyl-1 <i>H</i> -imidazol-3-ium bromide	Mild steel	1 M HCl (25–55°C)	17.0–19.1; 17.6–18.7	[66]
1-Decyl-3-methylimidazolium chloride	P110 steel	15% HCl (35°C)	24.1 (El-Awady adsorption isotherm)	[67]
4-Phenylimidazole	Copper (99.99% purity)	0.5 M H ₂ SO ₄ (25°C)	29.2	[68]
1-Allyl-3-ethylimidazolium bromide; 1-Allyl-3-butyylimidazolium bromide; 1-Allyl-3-hexylimidazolium bromide; 1-Allyl-3-octylimidazolium bromide	Copper (99.8% purity)	0.5 M H ₂ SO ₄ (25°C)	36–41	[69]
Imidazole; methyl imidazole	Aluminum (99.99% purity)	0.5 M HCl (30°C)	26.6; 29.5 (Frumkin adsorption isotherm)	[72]
Imidazolines				
<i>N</i> -(2-(2-tridecyl-4,5-dihydro-1 <i>H</i> - imidazol-1-yl)ethyl)tetradecanamide	St37 steel (procured from Erdemir Steel Co., Turkey)	15% HCl (25°C)	27.1	[75]
2-Methyl-4-phenyl-1-tosyl-4,5- dihydro-1 <i>H</i> -imidazole	P110 carbon steel	1 M HCl (60°C)	28.3	[76]

Inhibitor	Type of the metal	Type of the corrosive medium	Standard adsorption free energy ($-\Delta G_{\text{ads}}^*$), kJ/mol	Ref.
2-(2-Tribromomethyl-4,5-dihydroimidazol-1-yl)-ethylamine	Mild steel	1 M HCl (30°C)	38.9	[78]
2-(2-Trifluoromethyl-4,5-dihydroimidazol-1-yl)-ethylamine; 2-(2-trichloromethyl-4,5-dihydroimidazol-1-yl)-ethylamine	Mild steel	1 M HCl (30°C)	34.7; 39.7	[79]
Imidazolidines				
3-Methyl-5,5'-diphenylimidazolidine-2,4-dione	Mild steel	1 M HCl (25°C)	40.3	[80]
Pyrazoles				
2-(Bis((1 <i>H</i> -pyrazol-1-yl)methyl)amino)pyrimidine-4,6-diol	Mild steel	1 M HCl (25°C)	40.2	[82]
1,5-Dimethyl-1 <i>H</i> -pyrazole-3-carbohydrazide	Mild steel	1 M HCl (35°C)	41.8	[84]
N1,N1-Bis(2-(bis((3,5-dimethyl-1 <i>H</i> -pyrazol-1-yl) methyl) amino)ethyl)-N2,N2-bis((3,5-dimethyl-1 <i>H</i> -pyrazol-1-yl) methyl)ethane-1,2-diamine; diethyl 1,1'-(((4-acetylphenyl) azanediyl)-bis(methylene))-bis(5-methyl-1 <i>H</i> -pyrazole-3-carboxylate)	Mild steel	1 M HCl (35°C)	41.8	[85]
(<i>E</i>)-N'-Benzylidene-2-(3,5-dimethyl-1 <i>H</i> -pyrazol-1-yl)acetohydrazide; (<i>E</i>)-N'-(4-chlorobenzylidene)-2-(3,5-dimethyl-1 <i>H</i> -pyrazol-1-yl)acetohydrazide	Mild steel	1 M HCl (35°C)	40.2; 43.2	[86]
(<i>E</i>)-5-(4-(Dimethylamino)phenyl)-3-(4-(dimethylamino)styryl)-2,3-dihydro-1 <i>H</i> -pyrazole-1-carbothioamide	Mild steel	1 M HCl (30–60°C)	43.0–44.7	[87]
2-(3-Methyl-1 <i>H</i> -pyrazol-5-yl)pyridine	Mild steel	1 M HCl (35°C)	42 (Frumkin adsorption isotherm)	[88]

Inhibitor	Type of the metal	Type of the corrosive medium	Standard adsorption free energy ($-\Delta G_{\text{ads}}^*$), kJ/mol	Ref.
(<i>E</i>)-5-Amino-3-(4-methoxyphenyl)- <i>N'</i> -(1-(4-methoxyphenyl)ethylidene)-1 <i>H</i> -pyrazole-4-carbohydrazide; (<i>E</i>)-5-amino- <i>N'</i> -(4-chlorobenzylidene)-3-(4-chlorophenyl)-1 <i>H</i> -pyrazole-4-carbohydrazide	Mild steel	15% HCl (30–60°C)	36.8–39.9; 36.5–39.3	[89]
1-{[Benzyl-(2-cyano-ethyl)-amino]-methyl}-5-methyl-1 <i>H</i> -pyrazole-3-carboxylic acid ethyl ester	Mild steel	1 M HCl (35–70°C)	37.1–42.0	[90]
<i>N</i> 1, <i>N</i> 1, <i>N</i> 3, <i>N</i> 3-Tetrakis((3,5-dimethyl-1 <i>H</i> -pyrazol-1-yl)methyl)propane-1,3-diamine; <i>N</i> 1, <i>N</i> 1, <i>N</i> 2, <i>N</i> 2-tetrakis-((3,5-dimethyl-1 <i>H</i> -pyrazol-1-yl)methyl)benzene-1,2-diamine	Mild steel	1 M HCl (40–70°C)	38.4–39.3; 39.3–40.1	[91]
3'-(3,4-Dichlorophenyl)-2',3',6,7,8,9-hexahydro-2'-phenyl-5'-styrylspiro[benzocyclo-heptene-6(5 <i>H</i>),4'-(4 <i>H</i> -pyrazol)]-5-one	Copper	2 M HNO ₃ (25–50°C)	10.3–11.4 (Freundlich adsorption isotherm)	[95]
Pyrazolines				
[5-Hydroxy-3-phenyl-5-(phenylamino)-4,5-dihydro-1 <i>H</i> -pyrazol-1-yl](phenyl)methanone; (2-hydroxyphenyl)[5-hydroxy-3-phenyl-5-(phenylamino)-4,5-dihydro-1 <i>H</i> -pyrazol-1-yl]methanone; (2-aminophenyl)[5-hydroxy-3-phenyl-5-(phenylamino)-4,5-dihydro-1 <i>H</i> -pyrazol-1-yl]methanone; 5-hydroxy-3-phenyl-5-(phenylamino)-4,5-dihydro-1 <i>H</i> -pyrazole-1-carbothioamide	Mild steel	1 M H ₂ SO ₄ (30°C)	26.2; 28.6; 30.5; 30.3	[96]
2-(4-(5-(<i>p</i> -Tolyl)-4,5-dihydro-1 <i>H</i> -pyrazol-3-yl)phenoxy)acetic acid; 2-(4-(5-(4-nitrophenyl)-4,5-dihydro-1 <i>H</i> -pyrazol-3-yl)phenoxy)acetic acid	Mild steel	1 M HCl (30°C)	36; 37	[97]

Inhibitor	Type of the metal	Type of the corrosive medium	Standard adsorption free energy ($-\Delta G_{\text{ads}}^*$), kJ/mol	Ref.
2-(4-(4,5-Dihydro-3-(4-methoxyphenyl)-1H-pyrazol-5-yl)phenoxy)acetic acid; 2-(4-(4,5-dihydro-3-p-tolyl-1H-pyrazol-5-yl)phenoxy)acetic acid	Mild steel	1 M HCl (30°C)	34.4; 33.9	[98]
5-(4-(Dimethylamino)phenyl)-3-phenyl-4,5-dihydro-1H-pyrazole-1-carboxamide; (E)-5-(4-(dimethylamino)phenyl)-3-(4-(dimethylamino)styryl)-4,5-dihydro-1H-pyrazole-1-carboxamide	Mild steel	1 M HCl (30–60°C)	33.8–36.3; 34.6–40.0	[99]
5,5'-(1,4-Phenylene)-bis-[1-formyl-4,5-dihydro-3-phenyl-1H-pyrazole]	Carbon steel	1 M HCl (60°C)	34.7	[100]
1,2,3-Triazoles				
(1-p-Tolyl-1H-1,2,3-triazol-4-yl) methanol	Mild steel	1 M HCl (25–60°C)	46.2–50.2 (Temkin adsorption isotherm)	[103]
(1-(4-Aminophenyl)-1H-1,2,3-triazol-4-yl)methanol; 1-(1-(4-aminophenyl)-5-methyl-1H-1,2,3-triazol-4-yl)ethanol	Carbon steel	1 M HCl (25°C)	33.6; 33.1	[104]
(R)-Benzyl-2-amino-3-((1-(((2R,3R,4S,5R,6S)-3,4,5-tris(benzyloxy)-6-methoxytetrahydro-2H-pyran-2-yl)methyl)-1H-1,2,3-triazol-4-yl)methoxy)propanoate; (2R)-benzyl-2-amino-3-((1-(((2R,3R,4S,5R,6S)-3,4,5-tris(benzyloxy)-6-methoxytetrahydro-2H-pyran-2-yl)methyl)-1H-1,2,3-triazol-4-yl)methoxy)butanoate; (2R)-benzyl-2-amino-3-((1-(((2R,3R,4S,5S,6S)-3,4,5-tris(benzyloxy)-6-methoxytetrahydro-2H-pyran-2-yl)methyl)-1H-1,2,3-triazol-4-yl)methoxy)butanoate	Mild steel	1 M HCl (25°C)	37.6; 38.4; 37.7	[105]

Inhibitor	Type of the metal	Type of the corrosive medium	Standard adsorption free energy ($-\Delta G_{\text{ads}}^*$), kJ/mol	Ref.
1-Benzyl-4-phenyl-1 <i>H</i> -1,2,3-triazole	Mild steel	1 M HCl (25–55°C)	35.7–39.8	[106]
(1-Benzyl-1 <i>H</i> -1,2,3-triazole-4-yl)methanol; (1-(pyridin-4-ylmethyl)-1 <i>H</i> -1,2,3-triazole-4-yl)methanol	Mild steel	1 M HCl (25°C)	31.3; 36.0	[107]
4-Amino-1-((8-hydroxyquinolin-5-yl)methyl)-1,2,3-triazole-5-carboxylate	Carbon steel	1 M HCl (25°C)	43	[108]
1-(5-Methyl-1-phenyl-1 <i>H</i> -1,2,3-triazol-4-yl)ethanone	Mild steel	1 M HCl (30°C)	39.9	[109]
4,5-Diethyl-1-[(4-ethyl-2-phenyl-4,5-dihydro-1,3-oxazol-4-yl)methyl]-4,5-dihydro-1 <i>H</i> -1,2,3-triazole-4,5-dicarboxylate	Mild steel	1 M HCl (25°C)	42.8	[110]
1,2,4-Triazoles				
3,5- <i>Bis</i> (<i>R</i>)-4-amino-1,2,4-triazole (<i>R</i> = 4-methoxyphenyl; 2-thienylmethyl)	Mild steel, carbon steel	1 M HCl (30°C)	40.8; 45.7	[111, 114]
3,5- <i>Bis</i> (<i>R</i>)-4-amino-1,2,4-triazole (<i>R</i> = 4-methoxyphenyl; 4-chlorophenyl; 4-tolyl; 3,4-dimethoxyphenyl)	Mild steel	2 M H ₃ PO ₄ (35°C)	41.1; 39.8; 39.8; 40.0	[116, 117]
4-(4-Hydroxy-3-methoxybenzylideneamino)-4 <i>H</i> -1,2,4-triazole-3,5-dimethanol	Mild steel	0.5 M HCl (30–60°C)	31.9–36.2	[118]
(3-Phenylallylidene) amino-5-(pyridine-4-yl)-4 <i>H</i> -1,2,4-triazole-3-thiol; 3-mercapto-5-(pyridine-4-yl)-4 <i>H</i> -1,2,4-triazole-4-yl)imino)methyl)phenol; (4-nitrobenzylidene) amino)-5-(pyridine-4-yl)-4 <i>H</i> -1,2,4-triazole-3-thiol	Mild steel	1 M HCl (35–65°C)	32.3–37.0; 32.0–35.0; 31.6–33.4	[119]

Inhibitor	Type of the metal	Type of the corrosive medium	Standard adsorption free energy ($-\Delta G_{\text{ads}}^*$), kJ/mol	Ref.
(3-Bromo-4-fluoro-benzylidene)-[1,2,4]triazol-4-yl-amine; (4-trifluoromethyl-benzylidene)-[1,2,4]triazol-4-yl-amine; (2-fluoro-4-nitro-benzylidene)-[1,2,4]triazol-4-yl-amine	Mild steel	0.5 M HCl (30–60°C)	31.1–34.1; 31.0–33.5; 30.5–33.0	[119]
4-Salicylideneamino-3-phenyl-5-mercapto-1,2,4-triazole	Mild steel	1 M HCl (25–70°C)	32.8–36.1	[121]
5-((Furan-2-yl)methyleneamino)-2H-1,2,4-triazole-3-thiol; 5-((thiophen-2-yl)methyleneamino)-2H-1,2,4-triazole-3-thiol	Mild steel	1 M HCl (27°C)	34.3; 35.6	[123]
3,5-Bis(methylene octadecyl dimethylammonium chloride)-1,2,4-triazole	Carbon steel	1 M HCl (25°C)	42.1	[128]
5-Hexylsulfanyl-1,2,4-triazole	Carbon steel	1 M HCl (25°C)	45.9	[129]
5-Octylsulfanyl-1,2,4-triazole; 5-decylsulfanyl-1,2,4-triazole	Mild steel	1 M HCl (30°C)	41.4; 42.2	[130]
4-{[4-(Dimethylamino)benzylidene]-amino}-5-methyl-4H-1,2,4-triazole-3-thiol	316 Stainless steel	2 M HCl (30–60°C)	30.9–38.9	[139]
4-Amino-4H-1,2,4-triazole-3thiol; 4-amino-5-methyl-4H-1,2,4-triazole-3thiol; 4-amino-5-ethyl-4H-1,2,4-triazole-3thiol	Copper (99.9%)	0.5 M HCl (30°C)	36.5; 37.3; 37.7	[145]
3,5-Bis(4-methoxyphenyl)-4-amino-1,2,4-triazole	Copper (99.9%)	1 M HCl (30°C)	42.9	[147]
1,2,3,4-Tetrazoles				
3,3'-(3,3'-Dimethoxy[1,1'-biphenyl]-4,4'-diyl)-bis(2,5-diphenyl-2H-tetrazolium) dichloride	Cold rolled steel	1 M HCl (20°C)	40.7	[151]

Inhibitor	Type of the metal	Type of the corrosive medium	Standard adsorption free energy ($-\Delta G_{\text{ads}}^*$), kJ/mol	Ref.
(<i>E</i>)-3-Phenyl-2-(1 <i>H</i> - tetrazole-5-yl)acrylonitrile; (<i>E</i>)-3-(4-nitrophenyl)-2-(1 <i>H</i> - tetrazole-5-yl)acrylonitrile; (<i>E</i>)-3-(4-hydroxyphenyl)-2-(1 <i>H</i> - tetrazole-5- yl)acrylonitrile	Mild steel	1 M HCl (35–65°C)	34.9–36.7; 36.2–38.2; 37.6–39.7	[152]
2,3,5-Triphenyltetrazolium chloride	Cold rolled steel	1 M H ₂ SO ₄ (20–50°C); 7 M H ₂ SO ₄ (20°C)	29.4–32.5; 34.5	[155, 159]
2,2'-Bis(4-nitrophenyl)-5,5'-diphenyl-3,3'-(3,3'-dimethoxy-4,4'-diphenylene)ditetrazolium chloride	Cold rolled steel	0.5 M H ₂ SO ₄ (25°C)	41.0	[156]
3-(4,5-Dimethyl-2-thiazolyl)-2,5-diphenyl-2 <i>H</i> -tetrazolium bromide	Cold rolled steel	1 M HCl (30°C); 0.5 M H ₂ SO ₄ (30°C)	36.5; 33.7	[157]
3,3'-(3,3'-Dimethoxy[1,1'-biphenyl]-4,4'-diyl)-bis(2,5-diphenyl-2 <i>H</i> -tetrazolium) dichloride	Cold rolled steel	1 M H ₂ SO ₄ (20°C)	39.1	[158]
5-Mercapto-1-methyltetrazole; 1-[2-(dimethylamino)ethyl]-1 <i>H</i> -tetrazole-5-thiol; 1-(4-hydroxyphenyl)-1 <i>H</i> -tetrazole-5-thiol	X80 Steel	0.5 M H ₂ SO ₄ (25°C)	34.3; 33.7; 32.7	[161]
3,3'-(3,3'-Dimethoxy[1,1'-biphenyl]-4,4'-diyl)-bis(2,5-diphenyl-2 <i>H</i> -tetrazolium) dichloride; 2,2'-bis(4-nitrophenyl)-5,5'-diphenyl-3,3'-(3,3'-dimethoxy-4,4'-diphenylene)ditetrazolium chloride;	Cold rolled steel	2.5 M H ₃ CCOOH (20°C)	37.4; 38.7	[163]

Inhibitor	Type of the metal	Type of the corrosive medium	Standard adsorption free energy ($-\Delta G_{\text{ads}}^*$), kJ/mol	Ref.
3-(4,5-Dimethyl-2-thiazolyl)-2,5-diphenyl-2 <i>H</i> -tetrazolium bromide	Cold rolled steel	1 M ClCH ₂ COOH; 1 M Cl ₂ CHCOOH; 1 M Cl ₃ CCOOH (25°C)	33.1; 34.0; 34.9	[164]
5-Phenyltetrazole; 5-(2-bromophenyl)-1 <i>H</i> -tetrazole; 5-(4-bromophenyl)-2 <i>H</i> -tetrazole	Copper (99.99%)	0.5 M H ₂ SO ₄ (25°C)	38.2; 38.7; 37.8	[167]
1-Phenyl-1 <i>H</i> -tetrazole-5-thiol; 1-phenyl-1 <i>H</i> -tetrazole; 1 <i>H</i> -tetrazol-5-amine; 1 <i>H</i> -tetrazole	Aluminum	1 M HCl (25°C)	32.6; 32.1; 31.1; 28.8	[170]
Complex systems				
Indole	Mild steel Q235	0.1 M H ₂ SO ₄ (25–45°C)	28.5–28.8	[171]
5-Aminoindole; 5-chloroindole; indole	Mild steel	1 M H ₂ SO ₄ (25–55°C)	30.3–32.0; 24.8–28.0; 32.7–37.9	[172]
<i>N</i> -((1 <i>H</i> -Indol-3-yl)(phenyl)methyl)- <i>N</i> -ethylethanamine; 3-(phenyl(pyrrolidin-1-yl)methyl)-1 <i>H</i> -indole; 3-(phenyl(piperidin-1-yl)methyl)-1 <i>H</i> -indole	Mild steel	1 M HCl (35–65°C)	33.0–35.5; 33.7–36.4; 34.2–37.0	[174]
4-Hydroxy- <i>N'</i> -[(<i>E</i>)-(1 <i>H</i> -indole-2-yl)methylidene]]benzohydrazide	Mild steel	1 M HCl (30–60°C)	39.8–43.9	[175]
Indole-3-acetic acid	Mild steel	0.5 M HCl (25°C)	28.4	[177]
Indole-5-carboxylic acid	Mild steel	0.5 M H ₂ SO ₄ (25–45°C)	23.8–26.4	[178]
Indole-3-acetic acid	Copper (99.994%)	0.5 M H ₂ SO ₄ (25–55°C)	25.6–27.5	[179]

Inhibitor	Type of the metal	Type of the corrosive medium	Standard adsorption free energy ($-\Delta G_{\text{ads}}^*$), kJ/mol	Ref.
3,6-Dibromo-9-phenylcarbazole; 3-(9 <i>H</i> -carbazol-9-yl)-1,2-propanediol	Mild steel	1 M HCl (30°C)	23.1; 21.9 (Frumkin adsorption isotherm)	[180]
6-Methyl-2,3,4,9-tetrahydro-1 <i>H</i> - carbazole; 9-methyl-9 <i>H</i> -carbazole-3-carboxylic acid	Mild steel	1 M HCl (room <i>t</i>)	38.9; 37.4 (Frumkin adsorption isotherm)	[181]
9 <i>H</i> -Pyrido[3,4- <i>b</i>]indole; 1-methyl-9 <i>H</i> -pyrido[3,4- <i>b</i>]indole	C38 steel	1 M HCl (25°C)	18; 14	[182]
BI, 2-Methyl-BI, 5(6)-Carboxy-BI, 2- Amino-BI, 2-Hydroxymethyl-BI, 2- Mercapto-BI, 2-Acetonitrile-BI	Mild steel	1 M HCl (20°C)	23.2; 23.8; 28.0; 30.8; 31.0; 34.9; 39.7 (Frumkin adsorption isotherm)	[186]
2-Mercapto-BI	Mild steel	1 M HCl (40–80°C)	36.6–37.8	[187]
2-(2-Methyl-1 <i>H</i> -benzimidazol-1-yl) acethydrazide; 2-(2-ethyl-1 <i>H</i> -benzimidazol-1-yl) acethydrazide; 2-(2-propyl-1 <i>H</i> -benzimidazol-1-yl) acethydrazide	Mild steel	0.5 M HCl (30–50°C)	35.5–38.8; 37.2–39.9; 39.3–41.7	[188]
2-(Allylthio)-1 <i>H</i> -benzo[<i>d</i>]imidazole; <i>bis</i> ((1 <i>H</i> -benzo[<i>d</i>]imidazol-2- 4 yl)thio)methane	Mild steel	1 M HCl (25°C)	45.2; 46.3	[189]
1,4- <i>Bis</i> (2-(4-chlorophenyl)-1 <i>H</i> - benzo[<i>d</i>]imidazol-1-yl)butane; 1,4- <i>bis</i> (2-phenyl-1 <i>H</i> -benzo[<i>d</i>]imidazol- 1-yl)butane	Mild steel	1 M HCl (30°C)	35.3; 33.2	[190]
2-[(5-Methylpyrazol-3-yl)methyl] benzimidazole	C38 carbon steel	1 M HCl (25°C)	19.3	[192]
2-Aminomethylbenzimidazole; <i>bis</i> (2-benzimidazolylmethyl)amine; <i>tri</i> (2-benzimidazolylmethyl)amine	Mild steel	1 M HCl (25°C)	34.3; 37.0; 38.1	[193]

Inhibitor	Type of the metal	Type of the corrosive medium	Standard adsorption free energy ($-\Delta G_{\text{ads}}^*$), kJ/mol	Ref.
1-Butyl-2-(4-methylphenyl)benzimidazole	Mild steel	1 M HCl (30°C)	35.1	[194]
2-(1-(Morpholinomethyl)-1 <i>H</i> -benzo[d]imidazol-2-yl)phenol; 2-(1-((piperazine-1-yl)methyl)-1 <i>H</i> -benzo[d]imidazol-2-yl)phenol; 2-(1-((piperidine-1-yl)methyl)-1 <i>H</i> -benzo[d]imidazol-2-yl)phenol	N80 steel	15% HCl (30–60°C)	37.5–40.3; 37.3–40.1; 36.7–39.4	[195]
1-Butyl-2-thiobenzylbenzimidazole	Mild steel	1 M HCl (30–60°C)	45.5–46.2	[196]
3-(4-Chlorophenylacetylmethyl)-1-ethylbenzimidazolium tetrafluoroborate; 3-(4-chlorobenzoylmethyl)-1-methylbenzimidazolium bromide	Carbon steel	1 M HCl (30–60°C)	37.7–41.9; 32.4–39.5	[197, 198]
1-Butyl-3-methyl-1 <i>H</i> -benzimidazolium iodide	Mild steel	0.5 M H ₂ SO ₄ (25–55°C)	34.0–34.6	[199]
1,3-Bis[2-(4-methoxyphenyl)-2-oxoethyl]-1 <i>H</i> benzimidazol-3-ium bromide	6061 Al – 15 vol. pct. SiC _(p) composite	0.1 M HCl and 0.1 M H ₂ SO ₄ (30–50°C)	45.4–49.7; 37.1–40.8	[200]
6-Nitroso-2-phenylimidazo[1,2-a]pyridine-3 carbaldehyde; (2-phenylimidazo[1,2-a]pyridin-3-yl)methanol	Carbon steel	1 M HCl (25°C)	46.5; 44.1	[201]
4-Amino pyrazolo-pyrimidine; 3-bromine 4-amino pyrazolo-pyrimidine; 3-iodine 4-amino pyrazolo-pyrimidine; 4-hydroxypyrazolo-pyrimidine; 4-mercaptopyrazolo-pyrimidine	Pure copper	0.5 M H ₂ SO ₄ (25°C)	32.0; 32.4; 36.3; 35.9; 40.8	[202, 203]
1-Dodecyl-5-methyl-1 <i>H</i> -benzo[d][1,2,3]triazol-1-ium bromide	Carbon steel	7 M H ₃ PO ₄ (20–80°C)	37.2–41.6	[210]

*Adsorption energy data presented without additional clarification were calculated from the Langmuir isotherm.

More valuable and reliable data on NFHC adsorption on metals measured by direct methods are based on a decrease in the capacitance of the double electric layer (DEL) of a

steel electrode obtained by electrochemical impedance spectroscopy (EIS) and by the ellipsometry method. Studies of adsorption of a substituted triazole, IFKhAN-92, using the EIS method point to the chemisorption character of interaction between the molecules of this CI and the steel surface. At $t=22^{\circ}\text{C}$, the adsorption of IFKhAN-92 on a cathodically polarized low-carbon steel is described by Temkin's isotherm, while $(-\Delta G_{\text{ads}})$ is 42 kJ/mol in 2.0 M HCl [241], 49 kJ/mol in 2.0 M H_2SO_4 [242], and 51 kJ/mol in 2.0 M H_3PO_4 [243].

Importantly, the kinetics of IFKhAN-92 adsorption on low-carbon steel from mineral acid solutions is described by the Roginsky–Zeldovich equation characteristic of chemisorption processes. It was stated earlier that IFKhAN-92 is inefficient in steel protection in 2.0 M H_3PO_4 , but its combination with 0.5 mM KNCS allows the protection to be enhanced considerably. It was shown that addition of KNCS to 2.0 M H_3PO_4 has no effect on $(-\Delta G_{\text{ads}})$ of IFKhAN-92 on steel but accelerates its adsorption, as it is clearly demonstrated by the kinetic adsorption isotherms [243]. Apparently, this effect promotes accelerated formation of a protective CI layer that provides the required protection on the metal surface that otherwise quickly degrades in acid solutions. It is worth noting that this is only one of the possible impacts of the addition of KNCS on a corroding system.

EIS data point to the chemisorption interaction between IFKhAN-92 and the surface of chromium-nickel steels. In fact, the $(-\Delta G_{\text{ads}})$ values for IFKhAN-92 on cathodically polarized 12Kh18N10T steel calculated according to Temkin's equation are 55 ± 1 and 61 ± 1 kJ/mol in 2.0 M HCl and 2.0 M H_2SO_4 , accordingly [244]. This result agrees well with the data of ellipsometric studies of its adsorption, according to which the $(-\Delta G_{\text{ads}})$ value on this steel in 0.005–0.05 M HCl and 0.05 M H_2SO_4 is 55 ± 3 kJ/mol as calculated by Temkin's isotherm [245].

Important additional information on the nature of the bond between CI molecules and a metal surface can be obtained using quantum-chemical methods. In recent years, more and more studies have been appearing that employ quantum-chemical calculations to explain the inhibitory effect of NFHCs. Detailed analysis of the possibility of using quantum-chemical methods for explanation of the inhibiting effect of organic compounds, including NFHCs, is provided in reviews [246, 247], which allows us to leave this issue beyond the scope of our paper.

Valuable information on the composition and structure of protective layers formed by NFHCs on metal surfaces in acid media is provided by X-ray photoelectron spectroscopy (XPS) combined with layer-by-layer etching of a sample surface by argon ions. This method of XPS measurements allows one to determine the in-depth qualitative and quantitative composition of the protective layer. An important element of such studies is prolonged (up to 18 min) ultrasonic washing in distilled water or in acid solutions to remove the CIs from the surface of the metal sample. During this procedure, the CI molecules physically adsorbed on the metal surface are removed from the surface of samples pretreated in an inhibited acid solution. The CI molecules chemically bound to the metal surface are not removed by ultrasonic surface cleaning. Such metal samples demonstrate a protective aftereffect of CIs in

further corrosion tests in background solutions. The ultrasonic cleaning of the metal surface before XPS measurements provides important information proving the nature of the chemical bond between the CI molecules and the metal, since this method does not allow one to distinguish between CI particles present on the sample surface in the form of a protective layer and those remaining on the metal after incomplete removal of the inhibited corrosive medium.

Figure 3. XPS spectra of N1s (a) and Fe2p (b) electrons of surface of steel 3 after holding in 2 M HCl + 5 mM IFKhAN-92 (24 h, 20°C). With ultrasonic cleaning.

A protective layer with the simplest structure on the surface of steel 3 in 2 M HCl (22°C, 24 h) is formed by 4-substituted 1,2,4-triazole (IFKhAN-96) [136]. The adsorption of this compound on a steel surface leads to the formation of an apparent CI monolayer that is chemically bound to the metal surface. The organic CI monolayer is located over 4–7 nm thick iron(II) chloride and oxide phases directly adhering to the metal. A 3-substituted 1,2,4-triazole (IFKhAN-92) forms a more complex structure on steel 3 in an HCl solution [248, 249]. According to XPS data, a 4 nm thick protective layer of an organic CI (no more than 4 monomolecular layers) is formed on steel 3 samples pretreated in 2.0 M HCl + 5.0 mM IFKhAN-92 (20°C, 24 h). It includes an apparent monolayer of the organic CI no thicker than 2 nm that is directly chemisorbed on the metal sample surface and the overlying CI molecules physically bound to each other and to the chemisorbed layer. Physically bound CI layers are removed by sonication in water. The protective monolayer remaining on the metal surface in background 2 M HCl (20–60°C) manifests a protective aftereffect ($Z = 89–96\%$) that is weaker than in the case of a polymolecular CI layer ($Z = 95–99\%$). It is however sufficiently high to confirm the CI chemisorption on the steel surface (Table 3). The preservation of a CI monolayer on the metal surface after ultrasonic surface cleaning is also confirmed by an XP spectrum of N1s electrons of the steel surface showing different types of nitrogen atoms present in the structure of IFKhAN-92 molecules (Figure 3a). It is necessary to point out that, as shown by the spectra of Fe2p-electrons (Figure 3b), the metal surface under the CI layer is oxidized to iron oxides. A CI layer with a similar structure and similar protective properties is formed in 2.0 M H₂SO₄ + 5.0 mM IFKhAN-92 + 5.0 mM

KNCS (Table 3). However, despite the presence of KNCS in the inhibitory composition, no rhodanide anions are found in the protective layer.

Table 3. Corrosion rates (k , g/(m²·h)) and corrosion inhibition coefficients (γ) of steel 3 in 2 M HCl, H₂SO₄ and H₃PO₄ after holding (24 h) in inhibitor preliminary adsorption solution (room t). The duration of the experiments was 2 h. $t = 20$ –100°C.

Inhibitor preliminary adsorption solution	Solution for aftereffect study	k/γ	Temperature of background solutions, °C				
			20	40	60	80	100
—	2M HCl	k	1.44	4.71	33.4	204	902
Without ultrasonic washing							
2M HCl + 5.0 mM IFKhAN-92	2M HCl	k	0.018	0.24	0.44	1.03	554
		γ	80.0	19.6	75.9	198	1.6
After ultrasound washing							
2M HCl + 5.0 mM IFKhAN-92	2M HCl	k	0.16	0.39	1.2	136	—
		γ	9.0	12.1	27.8	1.5	—
—	2M H ₂ SO ₄	k	6.18	27.8	65.6	252	959
Without ultrasonic washing							
2M H ₂ SO ₄ + 5.0 mM IFKhAN-92 + 5.0 mM KNCS	2M H ₂ SO ₄	k	0.20	0.22	0.26	0.41	671
		γ	30.9	126	252	614	1.4
After ultrasonic washing							
2M H ₂ SO ₄ + 5.0 mM IFKhAN-92 + 5.0 mM KNCS	2M H ₂ SO ₄	k	0.25	0.66	0.97	201	—
		γ	24.7	42.1	67.6	1.3	—
—	2M H ₃ PO ₄	k	6.94	11.5	40.5	193	1020
Without ultrasonic washing							
2M H ₃ PO ₄ + 5.0 mM IFKhAN-92 + 5.0 mM KNCS	2M H ₃ PO ₄	k	0.054	0.31	0.52	2.54	1130
		γ	129	37.1	77.9	75.8	0.9
After ultrasonic washing							
2M H ₃ PO ₄ + 5.0 mM IFKhAN-92 + 5.0 mM KNCS	2M H ₃ PO ₄	k	0.064	1.07	46.3	—	—
		γ	108	10.7	0.89	—	—

Figure 4. XPS spectra of N1s (a) and Fe2p (b) electrons of surface of steel 3 after holding in 2 M H₃PO₄ + 5 mM IFKhAN-92 + 5 mM KNCS (24 h, 25°C).

The formation of protective CI layers with molecules bound by chemical bonds is observed on steel 3 in H₃PO₄ solutions [250]. Preadsorption of a mixture of IFKhAN-92 and KNCS on the steel surface from an H₃PO₄ solution results in the formation of a chemisorbed protective layer thicker than 4 nm. It has a polymeric nature and consists of a complex compound of IFKhAN-92 molecules, Fe(II) cations, and rhodanide anions. The formation of a protective layer is confirmed by the XP spectrum of N1s electrons of the steel surface obtained for nitrogen atoms of different types present in the structure of IFKhAN-92 molecules and rhodanide anions (Figure 4a). Such a layer is not removed from the steel surface in the course of ultrasonic cleaning and manifests a protective aftereffect in H₃PO₄ solutions (Table 3). As follows from the Fe2p spectra (Figure 4b), the metal surface underlying the CI layer is oxidized to iron oxides. Besides, the oxide phase includes a certain fraction of hydrogen phosphates. This follows from the presence of a peak of P2p electrons in the spectrum of the metal surface.

Figure 5. Protective after-effect of inhibitor layers formed on the surface of chromium-nickel steel 12Kh18N10T in 2 M HCl + 5 mM IFKhAN-92 (20°C) in 2 M HCl solution (2 h). 1 – samples after preliminary inhibitor adsorption (24 h), 2 – samples after preliminary inhibitor adsorption (24 h) followed by ultrasonic washing, 3 – samples without preliminary inhibitor adsorption.

Preliminary adsorption of IFKhAN-92 on the surface of 12Kh18N10T steel from HCl solution results in the formation of a polymolecular protective layer of the organic CI chemically bound to the phase of Fe, Cr, and Ni oxides and hydroxides adjoining the metallic phase [251, 252]. The lower part of the polymolecular protective layer of the organic CI consists of a polymeric complex formed by IFKhAN-92 molecules, metal cations (Fe, Cr, and Ni), and chloride anions, while the outer part consists of physically adsorbed IFKhAN-92 molecules. The weakly bound CI layers are removed from the metal surface in the course of its ultrasonic cleaning in HCl solution. The CI layer (about 3 monolayers) remaining on the metal surface manifests a protective aftereffect in 2 M HCl at t up to 80°C (Figure 5). This points to the chemical character of interaction between the organic CI within the layer of its complex polymer and the surface of the oxide–hydroxide phase. The formulation of IFKhAN-92 + urotropine (molar ratio 1:4) is more efficient in the protection of 12Kh18N10T steel. Studies of the surface composition of samples pretreated in such solutions show that urotropine, alongside with IFKhAN-92, is included in the chemisorbed organic CI layer. The amount of urotropine in the chemisorbed layer is in the range of 20–30%. The thickness of the protective layer formed in the presence of this CI formulation is smaller than in the case of individual IFKhAN-92. Strong protective properties at smaller thicknesses point to its more compact structure. This layer is strongly bound to the metal surface and is not removed after ultrasonic cleaning. The strong adhesion of IFKhAN-92 molecules and urotropine in the organic CI layer is explained by their chemical cross-linking that is initiated by a product on urotropine hydrolysis, formaldehyde.

Figure 6. Distribution of elements upon etching by argon ions of the surface of 12Kh18N10T steel exposed in 2 M H₂SO₄ + 4.0 mM IFKhAN-92 + 1.0 mM KNCS (24 h, 20°C).

Figure 7. Distribution of elements upon etching by argon ions of the surface of 12Kh18N10T steel exposed in 2 M H₂SO₄ + 2.5 mM IFKhAN-92 + 2.5 mM KI (24 h, 20°C).

We observed the formation of protective layers with a similar structure on a chromium-nickel steel protected in H₂SO₄ solutions by composite CIs: IFKhAN-92 + KNCS (Figure 6) and IFKhAN-92 + KI (Figure 7) [253, 254]. Protective layers of organic CI are chemically bound to the phases of Fe, Cr, and Ni hydroxides and oxides adjoining the metallic phase. The lower phase of the polymolecular protective layer of organic CI consists of a polymer complex formed by IFKhAN-92 molecules and metal (Fe, Cr, and Ni) cations, while the outer part is composed of physically adsorbed IFKhAN-92 molecules easily removed from the metal surface by sonication. In the case of the IFKhAN-92 + KNCS formulation, the polymer complex additionally contains rhodanide anions. The inclusion of bridging rhodanide anions into the layer provides the possibility of its thickening owing to the appearance of additional variants of coordination interaction of its particles. These protective layers are definitely close in structure to the polymeric layers of IFKhAN-92 formed on steel 3 in an H₃PO₄ + IFKhAN-92 + KNCS system. The protective layer formed by the formulation of IFKhAN-92 + KNCS has a thicker organic part but provides poorer protection than the layers formed by the formulation of IFKhAN-92 + KI. The probable cause lies in the composition and structure of the metal hydroxide/oxide layer. In the presence of KI, it is enriched with chromium that should improve its protective properties.

It was shown for 1,2,4-triazole derivatives that the efficient inhibition of metal corrosion by NFHCs in acid media is due to their ability to form strongly bound polymolecular protective layers on a metal surface. A particular feature of such layers is that the underlying organic CI layer is chemically bound to the metal surface. The CI layers above this layer contain molecules that are bound with each other either physically or chemically.

The maximum protective effects are provided by chemically bound CI layers. An oxide–hydroxide layer appears quite often between an organic CI layer and a metal surface. This additional layer makes its own contribution to the protective effect of the organic polymolecular layer. The chemical interaction of molecules in the protective polymeric layer is based on the coordination interaction of complexing agents (metal cations formed in the course of corrosion of the metal being protected) and ligands (molecules of nitrogen-containing heterocycles). The strongest protective layers are formed if the nitrogen-containing heterocycle is a bidentate ligand.

The role of additives enhancing the protective effect of NFHCs is to impart new properties to the protective layer in order to favor its best protective ability. The mechanisms of action of such additives are quite diverse. In fact, in the case of steel protection in acid solutions by a substituted triazole (IFKhAN-92), the rhodanide anion accelerates the adsorption of the organic CI on the metal. They simultaneously participate in the formation of a polymolecular protective film and are included in the structure of the polymeric complex. In the case of protection of alloys with a complex composition (stainless steels), iodide anions can change the composition of the oxide–hydroxide phase existing between the metal and the organic part of the protective layer. The urotropine additive is not only incorporated in the structure of a protective organic CI layer but also promotes the additional chemical binding of its constituent molecules of nitrogen-containing heterocycles.

There is vast literature data on the electrochemical behavior of NFHCs on metals in acid solutions, generally at temperatures close to room temperature. The results of voltammetric measurements show that these compounds efficiently inhibit cathodic and anodic reactions on metals. According to EIS studies, the addition of such CIs to a corrosive medium is accompanied by a decrease in the capacitance of the electric double layer on metals and an increase in the polarization resistance. The voltammetry and EIS data are also in good agreement with the corrosion rates of metal samples obtained by mass loss measurements.

Of special interest are electrochemical data corresponding to a wide range of temperatures, $t = 20\text{--}100^\circ\text{C}$. Formulations based on a substituted triazole (IFKhAN-92) can noticeably inhibit the electrode reactions of low-carbon steels in solutions of HCl, H_2SO_4 [220], and H_3PO_4 [255] and of chromium-nickel steels in solutions of HCl [140] and H_2SO_4 [143] even at t close to 100°C . A similar pattern was observed for low-carbon steel in acetic and formic acid solutions at $t = 100^\circ\text{C}$ [226], which is largely due to the formation of protective CI layers on metals by NFHCs.

Figure 8. Polarization curves of the steel 3 disk ($n = 460$ rpm) in 2.0 M H_3PO_4 (a), inhibited by 0.5 mM IFKhAN-92 + 0.5 mM KNCS (b) and 5.0 mM IFKhAN-92 + 0.5 mM KNCS (c), in the presence of FePO_4 (mol/L): 1 – 0.00, 2 – 0.01, 3 – 0.02, 4 – 0.05, 5 – 0.10. $t = 25^\circ\text{C}$.

A more complex mechanism of the protective action of NFHC-based composite CIs is implemented in the case of steel corrosion in acid media containing Fe(III) salts. Its understanding became possible owing to the employment of a complex of electrochemical research techniques: potentiometry, cyclic voltammetry on a platinum electrode, and voltammetry on a steel disk electrode. They were used to study both acid solutions containing Fe(III) salts and a (steel–acid solution) corrosion system. In H_3PO_4 solutions containing FePO_4 , the inhibitor formulation of IFKhAN-92 + KNCS efficiently inhibits the corrosion of low-carbon steels. In contrast, in H_2SO_4 solutions containing $\text{Fe}_2(\text{SO}_4)_3$ and in HCl solutions containing FeCl_3 , this CI has nearly no protective effect. The reason is that this CI efficiently inhibits the electrode reactions on steel only in H_3PO_4 solutions containing FePO_4 (Figure 8) [228].

In this system, the cathodic reaction of steel involves two partial processes, namely, the reduction of H^+ and Fe^{3+} . The first reaction occurs in the kinetic control zone, while the second one is controlled by diffusion. A rotating disk electrode is used to study electrochemical processes occurring with diffusion control. One can see that the rate of the cathodic reaction in an H_3PO_4 solution containing FePO_4 significantly depends on the disk electrode rotation rate (Figure 9). In a static solution, the cathodic reaction of steel is almost totally determined by the reduction of H^+ . Here, the rise on the cathodic current due to the electrode rotation is governed almost completely by the following reaction:

Figure 9. Dependence of the cathodic current density at $E = -0.30$ V on the square root of the rotational speed of the disk electrode (steel 3) in 2.0 M H_3PO_4 (a), inhibited by 0.5 mM IFKhAN-92 + 0.5 mM KNCS (b) and 5.0 mM IFKhAN-92 + 0.5 mM KNCS (c), in the presence of FePO_4 (mol/L): 1 – 0.00, 2 – 0.01, 3 – 0.02, 4 – 0.05, 5 – 0.10. $t = 25^\circ\text{C}$.

In a similar medium inhibited by IFKhAN -92 + KNCS, the cathodic current is much lower both in static and dynamic solutions. Moreover, the response cathodic current to a variation in the hydrodynamic mode of the solution is very weak. One can see that the IFKhAN-92 + KNCS formulation possesses a unique ability not only to suppress the reduction of H^+ but also to inhibit the reduction of Fe^{3+} considerably.

The protective layers formed by the IFKhAN-92 + KNCS formulation on a steel surface can provide efficient inhibition of Fe^{3+} reduction in H_3PO_4 solutions in the diffusion mode. This effect is, however, absent due to some reasons in H_2SO_4 and HCl solutions containing

Fe(III) salts. It has been shown using potentiometry [256, 257] and cyclic voltammetry [258, 259] that, as opposed to H_2SO_4 and HCl solutions, the oxidative ability of Fe(III) cations was lower in H_3PO_4 solutions owing to their complexation by phosphate anions. In addition, the mobility of Fe(III) cations decreased in H_3PO_4 solutions considerably [258, 259], which significantly affected the process of Fe(III) reduction occurring in the diffusion mode. Importantly, the presence of H_3PO_4 in H_2SO_4 and HCl solutions containing Fe(III) salts also removes the oxidative ability and mobility of Fe(III) cations in these solutions. This effect allows one to develop inhibited solutions of acid mixtures ($\text{H}_2\text{SO}_4 + \text{H}_3\text{PO}_4$ and $\text{HCl} + \text{H}_3\text{PO}_4$) that are stable to the accumulation of Fe(III), as discussed above.

A study on $\text{H}_2\text{SO}_4 + \text{H}_3\text{PO}_4$ and $\text{HCl} + \text{H}_3\text{PO}_4$ solutions containing Fe(III) salts demonstrated that the three-component formulation IFKhAN-92 + KNCS + urotropine had a higher efficiency in steel protection in these media than the binary formulation of IFKhAN-92 + KNCS. Here, the role of urotropine in this effect remained unclear. A potentiometric study of $\text{H}_2\text{SO}_4 + \text{H}_3\text{PO}_4$ and $\text{HCl} + \text{H}_3\text{PO}_4$ solutions containing Fe(III) salts showed that urotropine present in the solutions additionally bound Fe(III) cations into complex compounds, which resulted in an additional decrease in the oxidizing ability of these systems [260].

Thus, the development of inhibited acid solutions that are resistant to the accumulation of Fe(III) salts is possible only by combining the ability of H_3PO_4 -containing acid solutions to bind Fe(III) cations into complex compounds with lower corrosivity towards steels and the unique properties of the IFKhAN-92 + KNCS + urotropine formulation. This formulation not only forms a polymolecular protective CI layer of IFKhAN-92 molecules and rhodanide anions on a metal surface that efficiently suppresses Fe(III) reduction, but also, owing to the presence of urotropine, additionally decreases the oxidative ability of Fe(III) present in the corrosive medium.

Conclusion

So far, ample literature data have been accumulated regarding the protection of metals (predominantly steels) in NFHC-containing acid solutions. The studied CIs are both simple and complex heterocyclic systems including aromatic, unsaturated, and saturated ones. In these CI groups, the highest protective effect is often provided by heterocycle derivatives with bulky substituents. NFHCs tend to be adsorbed on metal surfaces from mineral acid solutions. One can assume with high probability, judging by the $(-\Delta G_{\text{ads}})$ values of these CIs on the metal surfaces, that some of them can be chemically bound to the metals. These compounds adsorbed on a metal surface inhibit the cathodic and anodic reactions, which ultimately suppresses its corrosion. An important feature of such compounds is their ability to form polymolecular protective layers on the metal surface, which allows these compounds to provide the most efficient protection.

An acceptable practical form of application of these compounds is to use them in order to develop composite CIs. NFHC-based formulations can be used for metal protection in acid media where a heterocycle alone is characterized by low efficiency. NFHC formulations

with some simple compounds (KI, KBr, KNCS, urotropine) can form protective polymolecular CI layers on a metal surface, where NFHC molecules are chemically bound with each other and with the metal surface being protected. They manifest high protection ability owing to the characteristic composition and structure of these layers. Owing to the ability to form such structures on the metal surface, composite NFHC-based CIs can protect various steels in very corrosive high-temperature solutions of mineral acids (t up to 200°C) and in Fe(III)-containing acid media.

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