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## SOME ACETOPHENONE DERIVATIVES AS CORROSION INHIBITORS

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**Abstract:** The inhibition activities of 2-hydroxy-5-methylacetophenone thiosemicarbazone (**1**), (Z)-2-(((E)-1-(2-hydroxy-5-methylphenyl)ethylidene)hydrazono)thiazolidin-4-one (**2**), (E)-3-(2-hydroxy-5-((E)-phenyldiazenyl)phenyl)-1-phenylprop-2-en-1-one (**3**), (E)-1-(4-bromophenyl)-3-(2-hydroxy-5-((E)-phenyldiazenyl)phenyl)prop-2-en-1-one (**4**), (E)-5-(2-hydroxy-5-(phenyldiazenyl)phenyl)-3-phenyl-4,5-dihydro-1H-pyrazole-1-carbothioamide (**5**), (E)-3-(4-bromophenyl)-5-(2-hydroxy-5-(phenyldiazenyl)phenyl)-4,5-dihydro-1H-pyrazole-1-carbothioamide (**6**), 2,2'-((1E,1'E)-(ethane-1,2-diylbis(azanylylidene))bis(methanylylidene))bis(4-((E)-phenyldiazenyl)phenol (**7**) and 2-((Z)-2-hydroxy-5-((E)-phenyldiazenyl)benzylidene)hydrazine-1-carbothioamide (**8**) were explored by means of weight loss measurements for mild St-3 specimen in brine-kerosene solution. The corrosion rates of the St-3 drops as the inhibitor concentration increases together with increase of inhibition efficiencies. Thermodynamic parameters of adsorption on the St-3 surface at different concentrations were calculated.

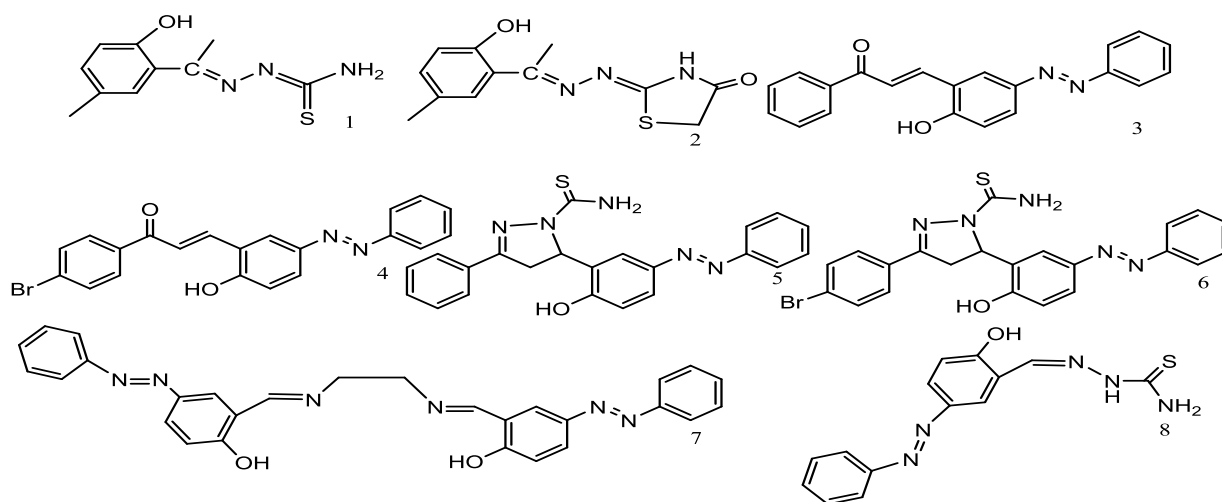
**Keywords:** adsorption, corrosion, surface, metal, nuclear magnetic resonance (NMR)

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## Introduction

The acetophenones and their derivatives are important intermediates in organic synthesis. They serve as starting material for the synthesis of variety of heterocyclic compounds of physiological significance. Because of the

presence of different functionalities, these compounds confer biological activities, such as antimicrobial, antibacterial, antifungal, anticancer, antitubercular, antiviral, antiinflammatory, and antihyperglycemic [1-12].



**Scheme.** The molecular structure of acetophenone derivatives

Also, acetophenones and their derivatives are known to act as corrosion inhibitors in aggressive media [13-17].

In the previous works we informed about the synthesis and NMR investigations of different acetophenone derivatives [18-28].

Our goal in this work was to examine inhibition activity of some acetophenone derivatives against corrosion of St-3 specimen immersed into the brine-kerosene solution. The molecular structure of investigated compounds is shown in Scheme.

### Results and discussion

Mild steel is a material frequently used in various areas of industry. But, in contact to the aggressive media, the corrosion process occurs on the steel surfacel. Different organic compounds were used successfully as inhibitors against the corrosion of different type steels and from year to year the interest is still growing in using other inhibitors. Among these compounds, acetophenone derivatives are known as inhibitors in aggressive media [13-17].

In this work, the gravimetric measurements are used for St-3 immersed brine-kerosene solution as analog of oil contaminated maritime water.

*Weight loss measurements.* The weight losses of St-3 specimens in brine-kerosene media (with/without adding different alcoholic solutions of analyzed inhibitors) are determined at 5 hour of immersion time and at 25<sup>0</sup>C. Values of CR, % IE are given in Table 1.

**Table 1.** Variation of corrosion rate\* (CR, mg/cm<sup>2</sup>·hour), inhibition efficiency (% IE) of the compounds (1-8) with their different concentrations in weight loss measurements at 300 min immersion time in 380 ml brine-kerosene-alcoholic solution and at 25<sup>0</sup>C

$C_{inh}, g$	Compound 1		Compound 2		Compound 3		Compound 4	
	CR	IE	CR	IE	CR	IE	CR	IE
0.005	$4.63 \cdot 10^{-5}$	49.56	$3.68 \cdot 10^{-5}$	63.73	$6.57 \cdot 10^{-5}$	64	$5.33 \cdot 10^{-5}$	71
0.01	$3.35 \cdot 10^{-5}$	60.10	$3.05 \cdot 10^{-5}$	79.5	$5.56 \cdot 10^{-5}$	70	$5.02 \cdot 10^{-5}$	73
0.03	$3.21 \cdot 10^{-5}$	68.31	$1.88 \cdot 10^{-5}$	91.56	$4.64 \cdot 10^{-5}$	75	$1.85 \cdot 10^{-5}$	90
$C_{inh}, g$	Compound 5		Compound 6		Compound 7		Compound 8	
	CR	IE	CR	IE	CR	IE	CR	IE
0.005	$1,11 \cdot 10^{-5}$	40	$3,94 \cdot 10^{-5}$	78	$4,61 \cdot 10^{-5}$	75	$6,03 \cdot 10^{-5}$	67
0.01	$9,5 \cdot 10^{-5}$	48	$3,79 \cdot 10^{-5}$	79	$3,94 \cdot 10^{-5}$	78	$3,55 \cdot 10^{-5}$	80
0.03	$8,26 \cdot 10^{-5}$	55	$3,7 \cdot 10^{-5}$	80	$1,08 \cdot 10^{-5}$	94	$2,47 \cdot 10^{-5}$	87

\*corrosion rate (CR, mg/cm<sup>2</sup>·hour), from weight loss measurements of St-3 specimens for blank solution at 300 min immersion time and at 25<sup>0</sup>C in brine-kerosene solution is  $5.41 \cdot 10^{-5}$ .

In all cases, increase of inhibitor concentration was accompanied by decrease in the

weight loss and rise in % IE. These results made it possible to infer that compounds 2, 4,

**6**, **7** and **8** (especially **2**, **4** and **7**, with their inhibition efficiency above 90%) under investigations are fairly efficient inhibitors for St-3 specimen in brine-kerosene solution.

Except, as seen from the Table 1, corrosion inhibition efficiency for the compound **7** is higher than other investigated compounds. Note that growing inhibition efficiency for the compound **7** probably is caused by the presence of two symmetrical sorption center and ethylene bridge in the investigated compound.

Also, we have seen from Table 1 that the corrosion inhibition efficiency for the compounds **1**, **3** and **5** is less than the compounds **2**, **4**, **6**, **7** and **8**. Note that the decrease of inhibition efficiency for these compounds is probably caused by the presence of steric factors in the compound **1** (in the previous work [26] we have reported about the possibility of the presence of conformational transitions for in the molecule of the compound **1** in solution) and absence of bromine atom in the **3** and **5**.

Acetophenone derivatives like **1-8** can inhibit the steel corrosion in brine-kerosene-solutions due to sorption centers in molecules. Indeed, as we have seen from the obtained re-

sults (Table 1), synthesized compounds showed higher inhibition efficiency. Thus, the increase of basicity, among other factors, would be expected to increase the inhibition efficiency. Thermodynamic parameters for adsorption of the compounds **1-8** on St-3 surface at 5 hour immersion time in brine-kerosene solution at different concentrations are given in Table 2.

It is well known that values of  $\Delta G_{ads}^0$  - 20 kJ/mol or lower indicate a physical adsorption while those more negative than -40 kJ/mol involve sharing or transfer of electrons from inhibitor molecules to the metal surface to form a coordinate type of bond (chemical adsorption). The possible adsorption mechanism are: (i) electrostatic interaction between the charged inhibitor molecules and charged steel surface (called as physical adsorption, (ii) direct adsorption on the basis of donor-acceptor interactions between the separate pairs of electrons of hetero-atoms, p-electrons of benzene and heterocyclic rings and the vacant d-orbitals of iron surface atoms (called as chemical adsorption [29, 30].

**Table 2.** Thermodynamic parameters for adsorption of **1-8** (0.005, 0.01, 0.03 g in 380 ml) on St-3 surface at 300 min immersion time in brine-kerosene-alcoholic solution at different concentrations

	$C_{inh}, g$				$C_{inh}, g$				$C_{inh}, g$				$C_{inh}, g$		
	0.005	0.01	0.03		0.005	0.01	0.03		0.005	0.01	0.03		0.005	0.01	0.03
<b>1</b>	0.005	$41 \cdot 10^3$	-28	<b>2</b>	0.005	$32 \cdot 10^3$	-32	<b>3</b>	0.005	$75 \cdot 10^3$	-35	<b>4</b>	0.005	$44 \cdot 10^3$	-32
	0.01	$35 \cdot 10^3$	-29		0.01	$33 \cdot 10^3$	-39		0.01	$41 \cdot 10^3$	-36		0.01	$9 \cdot 10^3$	-35
	0.03	$39 \cdot 10^3$	-30		0.03	$34 \cdot 10^3$	-40		0.03	$46 \cdot 10^3$	-37		0.03	$37 \cdot 10^3$	-41
<b>5</b>	0.005	$22 \cdot 10^3$	-34	<b>6</b>	0.005	$4 \cdot 10^3$	-30	<b>7</b>	0.005	$108 \cdot 10^3$	-36	<b>8</b>	0.005	$23 \cdot 10^3$	-34
	0.01	$68 \cdot 10^3$	-37		0.01	$14 \cdot 10^3$	-33		0.01	$64 \cdot 10^3$	-38		0.01	$15 \cdot 10^3$	-33
	0.03	$145 \cdot 10^3$	-38		0.03	$39 \cdot 10^3$	-35		0.03	$92 \cdot 10^3$	-42		0.03	$152 \cdot 10^3$	-39

As is seen from the Table 2, the  $\Delta G_{ads}^0$  value for the compounds **2**, **4**, **6**, **7** and **8** in high concentrations clearly indicates its chemical adsorption (-39, -40, -41 and -42 kJ/mol); however, in low concentrations it indicates complex sorption on the steel surface. The

range of calculated  $\Delta G_{ads}^0$  values for the compounds **1**, **3** and **5** vary between -30 and -38 kJ/mol. That means that adsorption of these inhibitors on the steel surface could involve complex interactions (physical adsorption and chemical adsorption).

## Conclusions

The explored compounds are efficient inhibitors (especially compounds **2**, **4** and **7**) against corrosion of St-3 in brine-kerosene solution as analog of aggressive oil contaminated in maritime water. The inhibitor efficiency increased at higher concentrations of inhibitors and good inhibition effect is observed at indicated concentration (30 mg in 380 ml or 78.95

1000 ml). Rise in inhibition efficiency for the compound **7** is probably is caused by the presence of symmetrical sorption center and ethylene bridge in the analyzed molecule.

Obtained results show that the analyzed compounds are good inhibitors in aggressive medium.

## Experimental

All the chemicals were obtained from commercial sources (Aldrich) and used as received.

NMR experiments were performed on a BRUKER FT NMR spectrometer (UltraShield™ Magnet) AVANCE 300 (300.130 MHz for  $^1\text{H}$  and 75.468 MHz for  $^{13}\text{C}$ ) with a BVT 3200 variable temperature unit in 5 mm sample tubes using Bruker Standard software (TopSpin 3.1). The  $^1\text{H}$  and  $^{13}\text{C}$  chemical shifts were referenced to internal tetramethylsilane (TMS); the experimental parameters for  $^1\text{H}$ : digital resolution = 0.23 Hz, SWH = 7530 Hz, TD = 32 K, SI = 16 K,  $90^\circ$  pulse-length = 10  $\mu\text{s}$ , PL1 = 3 dB, ns = 1, ds = 0, d1 = 1 s; for  $^{13}\text{C}$ : digital resolution = 0.27 Hz, SWH = 17985 Hz, TD = 64 K, SI = 32 K,  $90^\circ$  pulse-length = 9  $\mu\text{s}$ , PL1 = 1.5 dB, ns = 100, ds = 2, d1 = 3 s. NMR-grade DMSO- $d_6$  (99.7 %, containing 0.3%  $\text{H}_2\text{O}$ ) and acetone- $d_6$  were used for the solutions of **1-8**.

The synthesis of **1-8** was prepared by well-known literature methods [31-33].

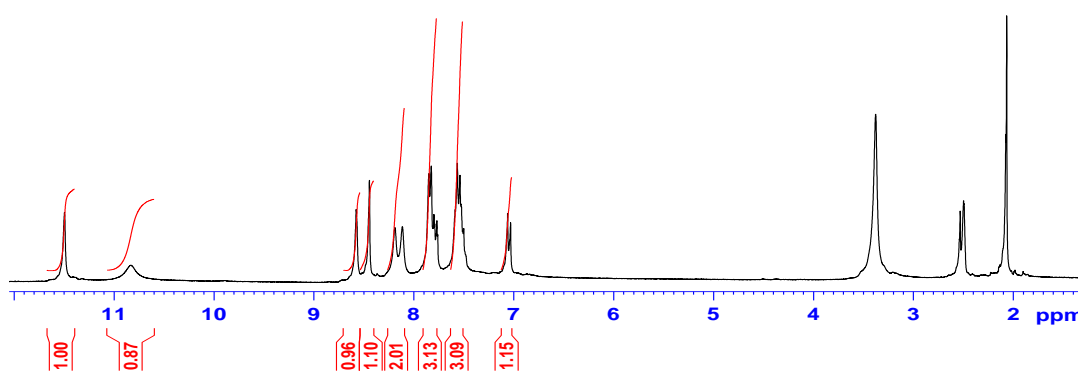
$^1\text{H}$  NMR spectrum of **1** (DMSO- $d_6$ ): 1.8 s (3H,  $\text{CH}_3$ ), 2.1 s (3H,  $\text{CH}_3$ ), 7.2-7.6 m (3H, arom.), 8.5 s (2H,  $\text{NH}_2$ ), 10.5 s (1H, OH), 11.2 s (1H, NH).

$^1\text{H}$  NMR spectrum of **2** (DMSO- $d_6$ ): 1.9 s (3H,  $\text{CH}_3$ ), 2.2 s (3H,  $\text{CH}_3$ ), 3.6 s (2H,  $\text{CH}_2$ ), 7.2-7.6 m (3H, arom.), 10.2 s (1H, NH), 10.7 s (1H, OH).

$^1\text{H}$  NMR spectrum of **3** (DMSO- $d_6$ ): 6.96 d (1H, arom.), 7.36-8.15 m (14H, arom. and  $\text{CH}=\text{CH}$ ).

$^1\text{H}$  NMR spectrum of **4** (DMSO- $d_6$ ): 7.1 d (1H, arom.), 7.3-8.2 m (13H, arom. and  $\text{CH}=\text{CH}$ ), 9.7s (1H, OH).

$^1\text{H}$  NMR spectrum of **5** (DMSO- $d_6$ ): 3.2-3.9 d (2H,  $\text{CH}_2$ ), 5.9 m (1H, CH), 6.7 d (1H, arom.), 7.1-7.8m (12H, arom.), 9.9s (1H, OH).



$^1\text{H}$  NMR spectrum of **8** in DMSO- $d_6$

<sup>1</sup>H NMR spectrum of **6** (DMSO-*d*<sub>6</sub>): 3.3-4.0 d (2H, CH<sub>2</sub>), 6.1m (1H, CH), 6.71 d (1H, arom.), 7.4-7.9 m (11H, arom.), 9.9 s (1H, arom.).

<sup>1</sup>H NMR spectrum of **7** (DMSO-*d*<sub>6</sub>): 3.7 s (2H, arom.), 6.9d (2H, arom.), 7.3-7.9 m (12H, arom.), 8.2 s (2H, arom.), 9.6 s (2H, CH=N), 10.9 s (2H, OH).

<sup>1</sup>H NMR spectrum of **8** (DMSO-*d*<sub>6</sub>): 6.9 d (1H, arom.), 7.2-7.4m (3H, arom.), 7.7-7.9 m (3H, arom.), 8.3 s (1H, arom.), 9.2 s (1H, CH=N), 9.8 s (1H, OH).

The composition (wt %) of steel-3 specimen used for all experiments is as follows: (0.14-0.22% C, 0.05-0.17% Si, 0.4-0.65% Mn, 0.3% Ni, 0.3%, Cu, 0.3% Cr, 0.08% As, 0.05% S and 0.04% P and the remainder iron), are abraded with different emery paper up to 1200 grade, washed thoroughly with bidistilled water, degreased and dried with acetone.

The aggressive corrosion medium is prepared in a 7:3 water/ethanol mixture (245:105 ml) for complete solubilization plus 30 ml kerosene and 12 g NaCl with the following inhibitor concentrations: 0 (blank), 0.005, 0.01, 0.03 g in 380 ml. Gravimetric measurements are carried out in terms of absence/presence of chalcones (or isoxazoles) in 500 ml glass flask supplied with a mixer. Finely abraded and dried mild steel specimens of dimension 2.39 cm × 1.89 cm × 0.37 cm were weighed on a digital balance with sensitivity of 0.00001g.

The effect of acetophenone derivative addition is tested at different concentrations on the corrosion of St-3 in brine-kerosene solution and explored through weight loss analysis at 298 K after 5 h of immersion period.

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### **BƏZİ ASETOFENON TÖRƏMƏLƏRİ KORROZIYA İNHİBİTORU KİMİ**

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*Təqdim olunan işdə 2-hidroksi-5-metilasetofenon tiösemiqarbazon (1), (Z)-2-(((E)-1-(2-hidroksi-5-metilfenil)etiliden)hidrazono)tiiazolidin-4-on (2), (E)-3-(2-hidroksi-5-((E)-fenildiazenil)fenil)-1-fenilprop-2-en-1-on (3), (E)-1-(4-bromfenil)-3-(2-hidroksi-5-((E)-fenildiazenil)fenil)prop-2-en-1-on (4), (E)-5-(2-hidroksi-5-(fenildiazenil)fenil)-3-fenil-4,5-dihidro-1H-pirazol-1-karbotioamid (5), (E)-3-(4-bromfenil)-5-(2-hidroksi-5-(fenildiazenil)fenil)-4,5-dihidro-1H-pirazol-1-karbotioamid (6), 2,2'-((1E,1'E)-(etan-1,2-diilbis(azanililiden))bis(meianililiden))bis(4-((E)-fenildiazenil)fenol (7) və 2-((Z)-2-hidroksi-5-((E)-fenildiazenil)benziliden)hidrazin-1-karbotioamidinin (8) duzlu su-kerosin məhlulunda polad-3 nümunəsinə qarşı korroziya inhibitoru xassəsi qravimetrik üsulla tədqiq edilmişdir. Müəyyən olunmuşdur ki, inhibitorun qatılığı artdıqca korroziyanın sürəti azalır, inhibitor effekti isə artır.*

*Açar sözlər: adsorbsiya, korroziya, səth, metal, nüvə magnit rezonansı (NMR)*

### **НЕКОТОРЫЕ ПРОИЗВОДНЫЕ АЦЕТОФЕНОНА В КАЧЕСТВЕ ИНГИБИТОРОВ КОРРОЗИИ**

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*Ингибиторная активность 2-гидрокси-5-метилацетофенон тиосемикарбазона (1), (Z) -2-(((E)-1-(2-гидрокси-5-метилфенил) этилиден) гидразон) тиазолидин-4-она (2), (E) -3- (2-гидрокси-5 - ((E) -*

фенилдиазенил)фенил) -1-фенилпроп-2-ен-1-она (3), (E) -1- (4-бромфенил) -3- (2-гидрокси-5 - ((E)-фенилдиазенил) фенил) проп-2-ен-1-она (4), (E) -5- (2-гидрокси-5- (фенилдиазенил)фенил) -3-фенил-4,5-дигидро-1H-пиразол-1-карботиоамида (5), (E)-3-(4-бромфенил)-5-(2-гидрокси-5- (фенилдиазенил)фенил)-4,5-дигидро-1H-пиразол-1-карботиоамида (6), 2,2'-((1E,1'E)-(этан-1,2-диилбис(азанилиден))бис(метанилиден))бис(4-((E)-фенилдиазенил)фенол (7) и 2-((Z)-2-гидрокси-5-((E)-фенилдиазенил)бензилиден)гидразин-1- карботиоамида (8) была исследована путем измерения потери веса для мягкого образца стали в солевом растворе вода-керосин. Обнаружено, что с увеличением концентрации ингибитора скорость коррозии  $S_t$ -снижается, а эффективность ингибирования увеличивается.

**Ключевые слова:** адсорбция, коррозия, поверхность, металл, ядерный магнитный резонанс (ЯМР)