# Protection of stainless steel in hydrochloric acid solution containing hydrogen sulfide by inhibitors

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### Abstract

Corrosion of chromium-nickel steel 1Kh18N9T in 2 M HCl at t = 20-100 °C was studied using the mass loss method. In parallel, hydrogen absorption by steel in the corrosive medium was measured by vacuum extraction method. IFKhAN-92 (a 1,2,4-triazole derivative) and its combination with urotropine were studied as corrosion inhibitors for stainless steel. It was shown that chromium-nickel steel 1Kh18N9T has low corrosion resistance in HCl solutions, particularly at temperatures close to 100°C. Under the conditions studied, the metal corrosion rate is k = 0.42 - 275 g/(m<sup>2</sup>·h) and increases with an increase in t. However, resistance of stainless steel to hydrogen absorption was noted. Addition of 15 mM H<sub>2</sub>S to the HCl solution considerably accelerates the corrosion of chromium-nickel steel ( $k = 3.2-800 \text{ g/(m^2 \cdot h)}$ ) and causes strong hydrogen absorption. The highest content of hydrogen absorbed in the metal (24 ml/100 g) was observed at  $t = 40^{\circ}$ C and 2 h exposure of the specimens. Longer exposures of steel specimens in the corrosive medium increase hydrogen absorption by the metal. Addition of IFKhAN-92 or a formulation of IFKhAN-92 with urotropine (at 1:4 molar ratio of the components) provides efficient inhibition of steel corrosion in hot HCl solutions ( $t = 80-100^{\circ}$ C). Corrosion is hindered 16–162 fold by IFKhAN-92 alone or 20–183 fold by its formulation with urotropine. We recommended the formulation of IFKhAN-92 with urotropine for protection of chromium-nickel steel in HCl solutions containing H<sub>2</sub>S. It efficiently hinders the metal corrosion (18–250 fold) up to 100°C and totally prevents hydrogen absorption up to 60°C, inclusively.

*Keywords:* acid corrosion, hydrogen sulfide, corrosion inhibitors, stainless steel, hydrogenation, triazoles.

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## Introduction

Hydrochloric acid treatment of gas/oil-bearing formations is an efficient method for treatment of bottom-hole areas in order to boost the extraction of liquid and gaseous

hydrocarbons [1, 2]. In the past decades, development of deep oil and gas fields with elevated temperatures and high hydrogen sulfide content has intensified. Treatment of these formations with hydrogen chloride involves saturation of the solution with hydrogen sulfide and an increase in solution temperature (t) [3–5], which increases considerably the acid corrosivity toward metal structures of gas- and oil-extraction equipment and adversely affects the protective capability of corrosion inhibitors added to HCl solutions [6, 7]. The presence of H<sub>2</sub>S in various mineralized aqueous media not only accelerates general corrosion of steels but also causes hydrogen absorption that impairs their mechanical properties [8].

It appears expedient to estimate the effect of hydrogen sulfide on the corrosion of chromium-nickel stainless steel in hydrogen chloride solution, both in the absence and in the presence corrosion inhibitors, in a broad temperature range. IFKhAN-92 (a 1,2,4-triazole derivative) and its formulation with urotropine were studied as corrosion inhibitors for chromium-nickel stainless steel. These inhibitors were chosen due to the fact that they can efficiently slow down the corrosion of chromium-nickel steel in the temperature range of  $t = 20-100^{\circ}$ C [9]. At the same time, it is known that IFKhAN-92 present in aqueous chloride media containing H<sub>2</sub>S ( $t = 25-80^{\circ}$ C) considerably suppresses the corrosion and hydrogen absorption of low-carbon and high-strength steels [10].

#### **Experimental procedure**

The corrosion rate of foil made of 1Kh18N9T stainless steel (composition, mass%: Fe – 71.92; Cr – 17.57; Ni – 9.16; Ti – 0.83; Si – 0.52) in 2 M HCl at  $t = 20-100^{\circ}$ C was determined from the mass loss of specimens (no less than 3 specimens per point) sized 20 mm × 10 mm × 0.25 mm, using 50 ml of the acid solution per specimen. The exposure time of the specimens in the solution was 0.25, 0.5, 1, or 2 h. Prior to an experiment, the specimens were cleaned with micron-graded polishing paper M40 and degreased with acetone.

Solutions were prepared from HCl of "chemically pure" grade and distilled water. HCl solutions were saturated with hydrogen sulfide ( $C_{H2S} = 15 \text{ mM}$ ) obtained from Na<sub>2</sub>S (technical grade, PRC standard: GB/T 10500–2000) and 15% H<sub>2</sub>SO<sub>4</sub> ("chemically pure" grade). The base concentrations (*C*) of individual inhibitors and their mixtures were 5.0 mM. A formulation containing 1 mM IFKhAN-92 + 4 mM urotropine was studied. As shown previously [9], this ratio of components is the best for protection of chromium-nickel stainless steels in HCl solutions.

The efficiency of inhibitors was estimated from the inhibition coefficient,  $\gamma = k_0/k_{in}$ , where  $k_0$  and  $k_{in}$  are the corrosion rates in the background solution and in the solution with the additive being studied, respectively. In the case of inhibited solutions containing hydrogen sulfide, 2 M HCl + 15 mM H<sub>2</sub>S was considered as the background solution. In

order to estimate quantitatively the effect of additives on nitrogen-containing inhibitors, the mutual influence coefficients of the mixture components were calculated [11]:

$$K_{\rm m} = \frac{\gamma_{\rm mix}}{\prod_{i=1}^{m} \gamma_i},$$

where  $\gamma_{\text{mix}}$  is the corrosion inhibition coefficient for the inhibitor mixture and  $\prod_{i=1}^{m} \gamma_i$  is the product of the corrosion inhibition coefficients for individual mixture components. If  $K_{\text{m}} < 1$ , the protection coefficients of the inhibitor components are mutually reduced; at  $K_{\text{m}} = 1$ , additive behavior is observed; and only at  $K_{\text{m}} > 1$  it should be concluded that the components of the inhibitor mixture mutually enhance protection.

The extent of hydrogen absorption by steel 1Kh18N9T was determined by vacuum extraction method. After corrosion tests, the specimens were placed in a vessel, air was evacuated from the vessel to a residual pressure of  $1.33 \cdot 10^{-4}$  Pa, and the vessel was heated to  $t = 500^{\circ}$ C. The amount of hydrogen evolved upon heating the specimens *in vacuo* was estimated from pressure change in 10 min ( $P_{tot}$ ) measured by a McLeod manometer at a constant volume of the evacuated part of the system. The pressure of evolved hydrogen ( $P_{H_2}$ ) was calculated from the change in total pressure ( $P_{tot}$ ) by the formula:

$$P_{\rm H_2} = P_{\rm tot} - P_{\rm nat},$$

where  $P_{\text{nat}}$  is the correction obtained in the blank test. The volume concentration of hydrogen in steel (ml/100 g of steel) was calculated by the formula:

$$V_{\rm H_2} = \frac{100 \, K P_{\rm H_2}}{M},$$

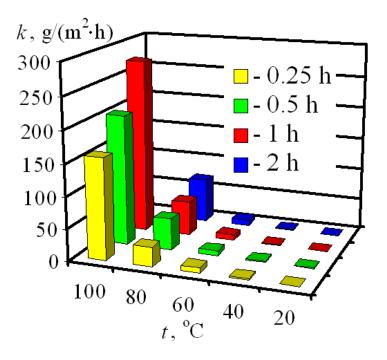
where K is a constant related to the volume of the analytical part of the set-up, and M is the mass of the steel specimen in grams.

The hydrogen content in the metal is given with correction for metallurgical hydrogen, the concentration of which in 1Kh18N9T steel amounts to 2.0 ml/100 g.

## Experimental results and discussion

Chromium-nickel steel is resistant to the action of cold 2 M HCl,  $k \le 0.67$  g/(m<sup>2</sup>·h) (Figure 1). However, the k value of steel gradually increases with a t increase. In fact, k increases 237-fold with a t increase by 80°C in short-term tests (0.25 h), or 640-fold upon longer exposure of specimens (1 h). Corrosion slows down with time at  $t \le 40$ °C but accelerates consistently at  $t \ge 60$ °C. Unfortunately, due to high k values at t = 100°C, we

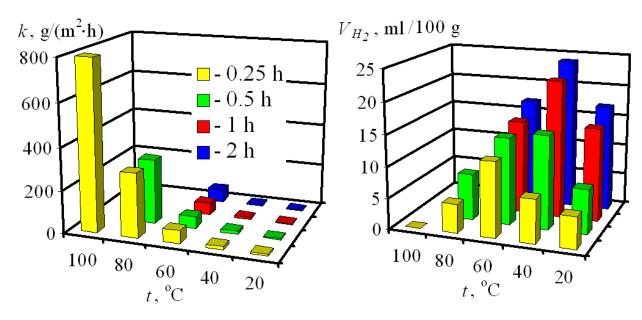
failed to obtain all the values within the selected time periods due to complete dissolution of the specimens. A similar situation was observed in H<sub>2</sub>S-containing acid solutions at  $t = 80^{\circ}$ C and especially at 100°C. In the entire *t* range studied, stainless steel in HCl solution containing no H<sub>2</sub>S does not undergo hydrogen absorption even after 2 h of exposure to the corrosive medium.



**Figure 1.** Corrosion rates of steel 1Kh18N9T in 2 M HCl as a function of specimen exposure time and solution temperature.

Addition of 15 mM H<sub>2</sub>S to the acid solution accelerates the metal corrosion considerably (Figure 2). For example, corrosion accelerates 7.6–11 fold at  $t = 20^{\circ}$ C. According to short-term tests (0.25 h), a *t* increase by 80°C results in a 111-fold increase in *k*. Corrosion slows down with time at  $t \le 40^{\circ}$ C but it nearly does not depend on exposure time at  $t \ge 60^{\circ}$ C.

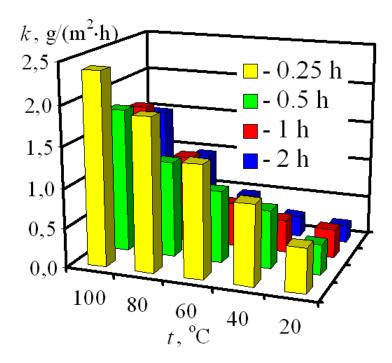
At  $t = 20^{\circ}$ C, stainless steel undergoes considerable hydrogen absorption in 2 M HCl + 15 mM H<sub>2</sub>S even after 0.25 h of exposure to the acid. Increasing the time of specimen exposure to the corrosive medium increases the hydrogen content in the metal 3.3-fold. Increasing the *t* of the solution results in an increase in the hydrogen content in steel. The maximum hydrogen absorption by the metal observed in the range of  $t = 40-60^{\circ}$ C changes to a decrease in hydrogen content, which becomes comparable to the metallurgical content at  $t = 100^{\circ}$ C. The highest content of hydrogen in steel (24 ml/100 g) was observed at  $t = 40^{\circ}$ C and 2 h exposure time of the specimens. In all the cases, longer exposures of steel specimens to the corrosive medium increase hydrogen absorption by the metal.



**Figure 2.** Corrosion rates of steel 1Kh18N9T in 2 M HCl + 15 mM  $H_2S$  and volume of hydrogen absorbed by the metal as a function of specimen exposure time and solution temperature.

The observed type of dependence of hydrogen content in stainless steel on the time of specimen exposure to the acid and on *t* can be explained by competition of two electrochemical processes: hydrogen evolution on the metal surface with incorporation of a fraction of hydrogen into the metal, and dissolution of a hydrogen-saturated surface layer of the metal. The first process favors hydrogen absorption by the metal, whereas on the contrary, the second one decreases the hydrogen content in the metal. Apparently, the first process mainly occurs at low *t* values and favors hydrogen absorption by the metal. An increase in *t* starts to enhance the second process considerably. Most likely, because of this the hydrogen content in steel decreases starting from  $t = 60^{\circ}$ C.

Addition of IFKhAN-92 alone to the HCl solution inhibits the metal corrosion only slightly (Figure 3, Table 1). It is only starting from  $t = 60^{\circ}$ C and 1 h exposure time that  $\gamma$  exceeds 10, which characterizes high efficiency of steel corrosion inhibition. The low protective effects of the inhibitor observed at  $t \le 60^{\circ}$ C are a consequence of metal corrosion resistance under these conditions resulting in low *k* values in the background acid solutions. In the acid solution inhibited with 5 mM IFKhAN-92, the maximum *k* is 0.55 g/(m<sup>2</sup>·h) at  $t = 20^{\circ}$ C, 1.0 g/(m<sup>2</sup>·h) at 40°C, and 1.4 g/(m<sup>2</sup>·h) at 60°C. At higher *t* values where steel corrosion in the background solution occurs with considerable *k* values, the  $\gamma$  values for steel are rather high. However, even at  $t = 100^{\circ}$ C, the maximum *k* in inhibited medium is 2.4 g/(m<sup>2</sup>·h). It should be noted that, like in the background HCl solution, exposure of steel in the acid containing the triazole studied does not result in hydrogen absorption by steel.



**Figure 3.** Corrosion rates of steel 1Kh18N9T in 2 M HCl containing 5 mM IFKhAN-92 as a function of specimen exposure time and solution temperature.

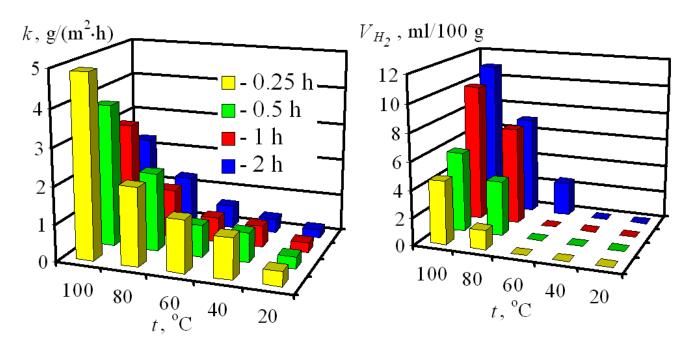
In the presence of 15 mM H<sub>2</sub>S in HCl solution inhibited with 5 mM IFKhAN-92 at  $t = 20^{\circ}$ C, the steel corrosion rate decreases to some extent in comparison with the acid solution in the absence of H<sub>2</sub>S (Figure 4). In contrast, corrosion is accelerated at higher temperatures. The *k* value increases 1.1–1.5 fold at  $t = 40^{\circ}$ C and 1.5–2.1 fold at  $t = 100^{\circ}$ C. Nevertheless, this medium is characterized by a high protective effect of the inhibitor ( $\gamma = 14-163$ ). This occurs because the *k* value in the background acid solution is rather high. The maximum *k* observed in the presence of 5 mM IFKhAN-92 was 4.9 g/(m<sup>2</sup>·h). In this case, no temperature maximum of the inhibition coefficient is reached for this additive, at least up to 100°C, which allows us to characterize it as a "high-temperature" corrosion inhibitor of stainless steel in HCl solution [12].

In 2 M HCl + 15 mM H<sub>2</sub>S, addition of 5 mM IFKhAN-92 prevents hydrogen absorption by stainless steel at  $t \le 60^{\circ}$ C. A hydrogen content in steel exceeding the metallurgical value is found only after 2 h exposure of the specimens at  $t = 60^{\circ}$ C. In hotter solutions ( $t = 80-100^{\circ}$ C), hydrogen absorption by the metal occurs, which increases with t increase and with time.

Thus, addition of IFKhAN-92 alone to an HCl solution containing H<sub>2</sub>S efficiently slows down the corrosion of 1Kh18N9T steel. Hydrogen absorption is totally suppressed at t = 20-40°C, as well as at t = 60°C at short exposures (up to 1 h) of the metal to the acid.

T 1914	_ 1	γvalues at various temperatures, °C				
Inhibitor	<i>τ</i> , h	20	40	60	80	100
Withou	ıt H <sub>2</sub> S					
	0.25	1.2	1.8	6.1	16	66
5 mM IFKhAN-92	0.5	1.2	2.1	9.0	42	114
	1	1.2	3.4	15	49	16
	2	2.0	6.2	19	71	_
	0.25	0.26	0.21	0.37	0.63	2.0
5 mM urotropine	0.5	0.13	0.12	0.25	0.91	1.8
	1	0.08	0.10	0.17	0.89	1.9
	2	0.05	0.09	0.15	0.77	_
	0.25	1.2	1.6	6.6	20	69
	0.5	1.1	2.0	8.7	45	13
1 mM IFKhAN-92 + 4 mM urotropine	1	1.5	2.8	11	49	18
	2	1.8	5.7	20	96	_
15 mM	$H_2S$					
	0.25	18	14	44	143	16
5 mM IEKh AN 02	0.5	16	16	67	145	_
5 mM IFKhAN-92	1	16	17	83	_	_
	2	15	21	101	_	
	0.25	6.0	3.3	6.5	11	12
5 mM urotroning	0.5	6.8	4.1	12	17	_
5 mM urotropine	1	6.7	4.6	20	_	
	2	6.0	6.8	44	_	
	0.25	24	27	48	143	25
$1 \text{ mM}$ IEV h AN 02 $\pm 4 \text{ mM}$ wrothering	0.5	20	26	57	235	
1 mM IFKhAN-92 + 4 mM urotropine	1	18	20	75	_	
	2	18	21	122	_	_

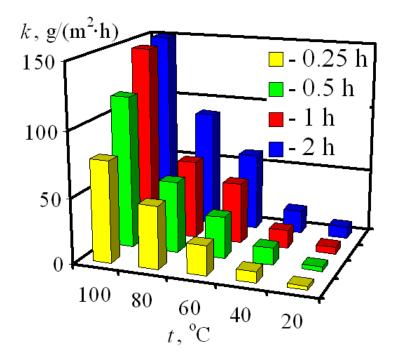
**Table 1.** Corrosion inhibition coefficients ( $\gamma$ ) of steel 1Kh18N9T in 2 M HCl provided by inhibitors at various temperatures and exposure times.



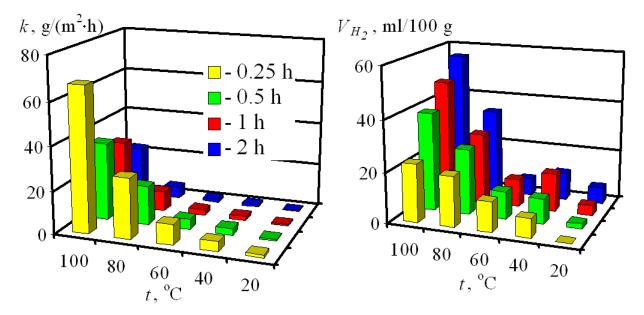
**Figure 4.** Corrosion rates of steel 1Kh18N9T in 2 M HCl + 15 mM  $H_2S$  containing 5 mM IFKhAN-92 and volume of hydrogen absorbed by the metal as a function of specimen exposure time and solution temperature.

Addition of 5 mM urotropine to HCl solution stimulates steel corrosion at  $t = 20-80^{\circ}$ C, and some corrosion is observed only at 100°C (Figure 5). In this case, the *k* of steel increases both with *t* increase and with time. However, in the entire *t* range studied, the hydrogen amount in steel remains at the metallurgical level after exposure of specimens to the urotropine-containing acid. We believe that the stimulating effect of urotropine results from the complexing capability of this compound or products of its acid degradation. Most likely, they are involved in the stage of metal anodic ionization, thus accelerating the anodic process and steel corrosion in general.

The presence of H<sub>2</sub>S beneficially affects the protective effect of urotropine (Figure 6). If it is present in solutions containing urotropine, the *k* values of steel are 2.2–17 times smaller at 20°C and 1.7–6.7 times smaller at 100°C in comparison with the same solution containing no hydrogen sulfide. In this case, the *k* of steel increases with *t* increase but strongly decreases with time. The *k* values obtained from 0.25 h and 2 h experiments differ 2.3–6.8 fold. Nevertheless, the protective effect of urotropine in this medium remains low ( $\gamma = 3.3-44$ ). The presence of H<sub>2</sub>S in this inhibited medium adversely affects hydrogen absorption by steel. The hydrogen content in steel can reach 56 ml/100 g.



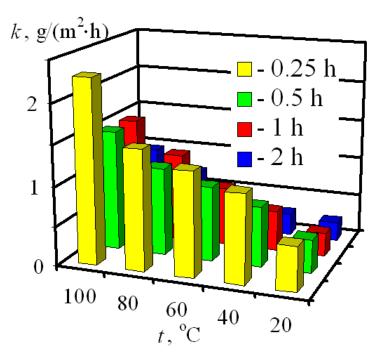
**Figure 5.** Corrosion rates of steel 1Kh18N9T in 2 M HCl containing 5 mM urotropine as a function of specimen exposure time and solution temperature.



**Figure 6.** Corrosion rates of steel 1Kh18N9T in 2 M HCl + 15 mM H<sub>2</sub>S containing 5 mM urotropine and volume of hydrogen absorbed by the metal as a function of specimen exposure time and solution temperature.

In 2 M HCl, the formulation containing 1 mM IFKhAN-92 and 4 mM urotropine is inferior in protective effect to 5 mM IFKhAN-92 at t = 20-40°C. Only at  $t \ge 80$ °C, the *k* values are lower for the formulation (Figure 7). Like for the triazole alone, the low

protective effects of the formulation observed on steel at  $t \le 60^{\circ}$ C result from low *k* values for the metal in the background HCl solution. Efficient inhibition of steel corrosion is observed only at  $t \ge 80^{\circ}$ C ( $\gamma = 20-183$ ). In general, the maximum *k* of steel observed in the presence of the inhibitor formulation was 2.3 g/(m<sup>2</sup>·h). It is interesting that the components of the formulation in question show a synergism of action in the absence of H<sub>2</sub>S (t = 20-80°C), which is due to the stimulating effect of urotropine on steel corrosion in HCl solution (Table 2). The hydrogen content in steel specimens that contacted 2 M HCl + 1 mM IFKhAN-92 + 4 mM urotropine remained at the metallurgical level.

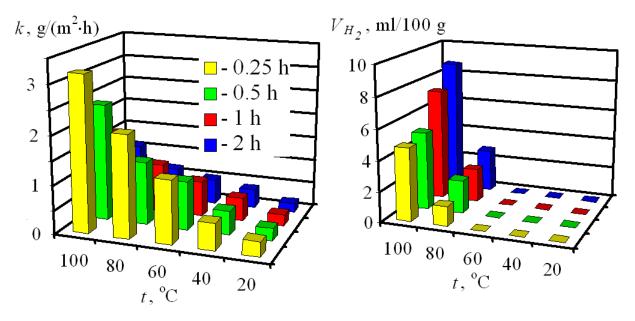


**Figure 7.** Corrosion rates of steel 1Kh18N9T in 2 M HCl containing 1 mM IFKhAN-92 + 4 mM urotropine as a function of specimen exposure time and solution temperature.

Addition of 15 mM H<sub>2</sub>S (Figure 8) beneficially affects the protective effect of the inhibitor formulation in the cold acid ( $t = 20^{\circ}$ C). In this case, k decreases 1.3–1.8 fold in comparison with the solution containing no hydrogen sulfide. In contrast, hydrogen sulfide accelerates corrosion at higher temperatures (80–100°C). In total, the inhibitive effects of the inhibitor formulation in 2 M HCl + 5 mM H<sub>2</sub>S are higher than those of the individual triazole, and the maximum k value is as small as 3.2 g/(m<sup>2</sup>·h). For example, even at 100°C the k value in the presence of the formulation is 1.5–2.1 times lower than that for IFKhAN-92. The inhibitor formulation slows down the metal corrosion with time. The  $\gamma$  coefficient of the mixture increases with an increase in t to 100°C, which is typical of a "high-temperature" inhibitor. In H<sub>2</sub>S-containing solutions, the effect of the inhibitor mixture components is antagonistic ( $K_m < 1$ ).

τ, h —	<i>K<sub>m</sub></i> values at various temperatures, °C						
	20	40	60	80	100		
		Wit	hout H <sub>2</sub> S				
0.25	3.8	4.2	2.9	2.0	0.52		
0.5	7.1	7.9	3.7	1.2	0.67		
1	17	8.2	4.3	1.1	0.59		
2	18	10	7.0	1.8	_		
		15	mM H <sub>2</sub> S				
0.25	0.22	0.58	0.17	0.09	0.13		
0.5	0.18	0.40	0.07	0.10	_		
1	0.17	0.26	0.05	_	_		
2	0.20	0.15	0.03	_	_		

**Table 2.** Mutual influence coefficients ( $K_m$ ) of inhibitor mixtures 1 mM IFKhAN-92 + 4 mM urotropine at various temperatures and exposure times.



**Figure 8.** Corrosion rates of steel 1Kh18N9T in 2 M HCl + 15 mM H<sub>2</sub>S containing 1 mM IFKhAN-92 + 4 mM urotropine and volume of hydrogen absorbed by the metal as a function of specimen exposure time and solution temperature.

The IFKhAN-92 + urotropine mixture totally prevents hydrogen absorption by steel up to  $t = 60^{\circ}$ C. However, the maximum hydrogen content is relatively small even at higher temperatures: 2.8 ml/100 g at 80°C and 8.7 ml/100 g at 100°C.

The optimum variant of inhibitor protection for 1Kh18N9T steel in HCl solution containing H<sub>2</sub>S (t = 20-100°C) is to use the IFKhAN-92 + urotropine combination (1:4 molar ratio of the components), which allows metal corrosion to be efficiently inhibited under these conditions and hydrogen absorption to be totally suppressed up to t = 60°C inclusively.

IFKhAN-92 and its combination with urotropine do not considerably lose their protective effect in HCl solution upon H<sub>2</sub>S addition that significantly increases the medium corrosivity toward steels. This feature in the behavior of these inhibitors opens new prospects of their practical application for protection of metals in mineral acids saturated with H<sub>2</sub>S. The results obtained in this study confirmed the previously noted versatility of triazole derivatives and formulations on their basis as corrosion inhibitors of various steels in highly corrosive acidic media [13], including high-temperature acid corrosion conditions [14, 15].

It can be assumed that efficient inhibition of corrosion and hydrogen absorption by chromium-nickel steels by the triazole derivative and formulation on its basis in HCl solutions, including those containing  $H_2S$ , is based on the unique capability of this inhibitor to undergo chemisorptive interaction with steel surface [16]. Strong adsorption of the triazole on stainless steel surface considerably hinders the electrode reactions occurring on its surface and eventually results in inhibition of the corrosion process in general [9]. Furthermore, the considerable suppression of the cathodic hydrogen evolution reaction by this inhibitor decreases hydrogen absorption by stainless steel [17].

#### Conclusions

- 1. The corrosion of chromium-nickel steel 1Kh18N9T in 2 M HCl in the range of  $t = 20 100^{\circ}$ C occurs at rates ranging within 0.42–275 g/(m<sup>2</sup>·h) and is accelerated with an increase in the temperature. Steel is resistant to hydrogen absorption under these conditions.
- 2. Addition of 15 mM H<sub>2</sub>S to 2 M HCl solution ( $t = 20-100^{\circ}$ C) considerably accelerates the corrosion of chromium-nickel steel 1Kh18N9T to rates ranging within 3.2–800 g/(m<sup>2</sup>·h). In this case, considerable hydrogen absorption by steel is observed.
- 3. Efficient corrosion inhibition of 1Kh18N9T steel in hot HCl solutions ( $t = 80-100^{\circ}$ C) can be achieved using IFKhAN-92 or a combination of IFKhAN-92 with urotropine (at 1:4 molar ratio of the components).
- 4. To protect chromium-nickel steel in HCl solution containing  $H_2S$ , we recommend the combination of IFKhAN-92 with urotropine (1:4) that efficiently inhibits steel corrosion up to 100°C and completely prevents hydrogen absorption up to 60°C inclusively.

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