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> APPLIED ELECTROCHEMISTRY AND CORROSION PROTECTION OF METALS

Allylacetylenes and Their Derivatives as Inhibitors of Steel Corrosion in Sulfuric Acid

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Abstract—New functionalized derivatives of allylacetylenes were prepared, and their ability to inhibit corrosion of St.3 low-carbon steel in 5 N sulfuric acid was studied. The effectiveness of inhibition of steel corrosion was studied in relation to the structure of acetylene derivatives, their concentration, and temperature of acidic medium.

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Functionalized unsaturated compounds are efficient inhibitors of the steel acid corrosion [1–4]. Acetylene derivatives strongly decelerate corrosion of steel in hydrochloric acid [5–9]. The multiple bonds with mobile π electrons determine high adsorption and protective properties of acetylene compounds. It was found that inhibition of the acid corrosion of steel with allylacetylenes only slightly depends on the nature of functional groups and substituents at unsaturated bonds; the decisive effect is exerted by acetylene and ethylene bonds.

Proceeding with studies of high-performance acid inhibitors, in this work we prepared new allylacetylenes and their derivatives and studied their inhibition properties in 5 M sulfuric acid in relation to the structure (nature of substituents).

Previously [10, 11] we studied the reaction of alkenyl halides with monosubstituted primary and tertiary acetylenic alcohols in the presence of EtMgBr/CuCl and NH₄Cl/CuCl, which are not universal reagents for preparing the corresponding allylacetylenic alcohols in a high yield. In this study, allylacetylenic alcohols I–VI were prepared by reaction of alkenyl halides with the corresponding monosubstituted acetylenylcarbinols in dimethylformamide (DMF) in the presence of copper iodide, triethylamine (TEA), and potassium carbonate:



where X = R = H(I); $X = H, R = CH_3(II)$; $X = CH_3$, R = H(III); $X = R = CH_3(IV)$; X = CI, R = H(V); $X = CI, R = CH_3(VI)$.

The other functionalized allylacetylene derivatives **VII–XXII** were prepared from appropriate alcohols **I–VI** in accordance with procedures given in [10–12] (Scheme 1), where X = R = H (**VII**), $X = CH_3$, R = H (**VIII, XI, XIV, XVII, XIX; X**) = Cl, R = H (**IX**); X = Cl, $R = CH_3$ (**X**); $X = R = CH_3$ (**XII, XV, XVIII, XX, XXI**); X = H, $R = CH_3$ (**XIII, XVI, XVIII, XXII**).

The physicochemical properties of compounds I-XXII are listed in Table 1. The structure and composition of the resulting compounds were studied by thinlayer (TLC) and gas-liquid (GLC) chromatography and by IR and ¹H NMR spectroscopy (Table 2) [10, 11, 13].

As seen from Scheme 1, the resulting allylacetylenes can be divided in two groups: (a) primary and tertiary alcohols of allylacetylene series and (b) ethers and esters of allylacetylene compounds, also containing functional groups.

When choosing the inhibitor structure, we took into account the fact that multiple (double and especially triple) bonds and functional groups $[OH, OCOCH_3, OCH(OC_2H_5)CH_3]$ provide efficient adsorption of the compounds on the steel surface with the formation of a protective film decelerating the corrosion.

EXPERIMENTAL

The IR spectra were recorded on a UR-20 spectrophotometer in the 4000-400 cm⁻¹ range (thin

Com-	Yield, %	bp, °C (P, mm Hg)	n_D^{20}	d_4^{20}	Found, %			Esemula	Calculated,		%
pound					С	Н	Cl(N)	Formula	С	Н	Cl(N)
I	74.2	80-81 (18)	1.4762	0.9373	74.81	8.46	_	C ₆ H ₈ O	74.97	8.38	_
II	80.7	70–71 (12)	1.4596	0.8997	77.50	9.63	-	$C_8H_{12}O$	77.37	9.74	—
III	82.9	92–94 (13)	1.4765	0.9284	76.46	9.30	-	$C_7H_{10}O$	76.32	9.15	—
IV	85.3	86.5-87 (13)	1.4625	0.8839	78.01	10.34	-	$C_9H_{14}O$	78.21	10.21	—
V	68.5	71–72 (0.5)	1.4820	1.0680	55.30	5.10	27.20	C ₆ H ₇ ClO	55.19	5.41	27.15
VI	78.6	64-65 (0.5)	1.4770	1.0428	60.45	6.80	22.20	$C_8H_{11}ClO$	60.55	6.99	22.34
VII	82.4	84-85 (10)	1.4540	0.9769	69.68	7.13	-	$C_8 H_{10} O_2$	69.54	7.30	—
VIII	85.8	95–96 (10)	1.4590	0.9700	71.18	7.79	-	$C_9H_{12}O_2$	71.02	7.95	—
IX	79.1	72–73 (0.5)	1.4720	1.1209	55.60	5.18	20.63	$C_8H_9ClO_2$	55.67	5.25	20.54
Χ	70.1	69–70 (0.5)	1.4660	1.0659	59.70	6.60	17.50	$C_{10}H_{13}ClO_2$	59.86	6.53	17.67
XI	75.2	60–61 (1)	1.4591	0.9287	72.55	9.86	-	$C_{11}H_{18}O_2$	72.48	9.95	—
XII	80.1	58–59 (1)	1.4572	0.9075	74.30	10.35	-	$C_{13}H_{22}O_2$	74.24	10.54	—
XIII	78.8	115–116 (1)	1.4767	1.0445	61.22	7.80	16.51	$C_{11}H_{17}ClO_2$	60.97	7.90	16.36
XIV	63.2	122–123 (1)	1.4894	1.0818	59.19	7.43	17.60	$C_{10}H_{15}ClO_2$	59.26	7.46	17.49
XV	67.1	123–124 (1.5)	1.4780	1.0293	62.39	8.19	15.55	$C_{12}H_{19}ClO_2$	62.46	8.30	15.36
XVI	85.2	78–79 (1)	1.4841	0.9539	73.40	8.92	-	$C_{11}H_{16}O_2$	73.30	8.94	-
XVII	66.4	98–99 (1.5)	1.4820	0.9843	72.19	8.55	-	$C_{10}H_{14}O_2$	72.26	8.49	_
XVIII	78.2	86-87 (1)	1.4665	0.9666	74.31	9.15	-	$C_{12}H_{18}O_2$	74.19	9.33	_
XIX	82.2	145–146 (1.5)	1.4878	0.9505	70.04	10.32	6.00	$C_{14}H_{23}NO$	70.19	10.44	5.85
XX	66.6	124–125 (1.5)	1.4674	0.9236	71.69	10.75	5.30	$C_{16}H_{28}NO$	71.80	10.84	5.23
XXI	72.1	104–105 (0.5)	1.4556	0.9467	71.20	9.71	-	$C_{15}H_{24}O_{3}$	71.39	9.59	-
XXII	76.1	89–90 (1)	1.4584	0.9620	70.68	9.29	-	$C_{14}H_{22}O_3$	70.55	9.30	-

Table 1. Properties of allylacetylenes and their derivatives I-XXII

Table 2. ¹H NMR and IR data for I-XXII

Com- pound	¹ Η NMR, δ, ppm	IR, v, cm^{-1}
V	5.20 s, 5.55 s (2H, $CH_2=C$), 3.10 s (2H, CH_2), 4.05 s (2H, CH_2O), 3.80 br s (1H OH)	630, 1395, 1650, 2230, 3100, 3350
VI	5.15 s, 5.45 s (2H, CH ₂ =C), 3.05 s (2H, CH ₂), 1.40 s [6H, (CH ₃) ₂], 3.90 br.s (1H, OH)	650, 1400, 1645, 2225, 3010, 3400
VII	4.90–5.43 m (2H, CH ₂ =C), 5.50–6.00 m (1H, C=CH), 2.85–3.05 m (2H, CH ₂), 4.50–4.70 m (2H, CH ₂ O), 2.00 s (3H, COCH ₃)	875, 990, 1160, 1248, 1656, 1746, 2235, 3010
VIII	4.75 s, 4.90 s (2H, CH ₂ =C), 1.75 s (3H, CH ₃), 2.85 s (2H, CH ₂), 4.55 s (2H, CH ₂ O), 2.10 s (3H, COCH ₃)	890, 910, 1175, 1645, 1740, 2240, 3100
IX	5.10 s, 5.60 s (2H, CH ₂ =C), 3.15 s (2H, CH ₂), 4.10 s (2H, CH ₂ O), 2.08 s (3H, COCH ₃)	640, 1170, 1246, 1380, 1656, 1745, 2235, 3090
X	5.15 s, 5.80 s (2H, CH ₂ =C), 3.10 s (2H, CH ₂), 1.53s [6H, (CH ₃) ₂], 1.96 s (3H, COCH ₃)	660, 1135, 1250, 1395, 1660, 1740, 2220, 3010
XI	4.70 s, 4.96 s (2H, $CH_2=C$), 1.80 s (3H, CH_3), 2.72 s (2H, CH_2), 4.45 s (2H, CH_2O), 4.40 q (1H, OCH), 3.26 q (2H, OCH_2), 1.15 d (3H, CH_3CH), 1.00 t (3H, CH_3CH_2)	885, 915, 1095, 1160, 1410, 1655, 2235,
XII	4.76 s, 4.87 s (2H, CH ₂ =C), 1.75 s (3 H, CH ₃), 2.86 s (2H, CH ₂), 1.40 s [6H, (CH ₃) ₂], 4.42 q (1H, OCH), 3.30 q (2H, OCH ₂), 1.17 d (3H, CH ₂ CH), 1.05 t (3H, CH ₂ CH ₂)	890, 920, 1100, 1175, 1645, 2240, 3100
XIII	4.90–5.40 m (2H, CH ₂ =C), 5.60–6.10 m (1H, C=CH), 2.75– 3.05 m (2H, CH ₂), 1.30 s [6H, (CH ₃) ₂], 3.30–3.70 m (4H, CH ₂ , CH ₂ Cl), 375–4.00 m (1H, CH–O), 2.35 br.s (1H, OH)	725, 880, 910, 1170, 1670, 2235, 3100, 3400

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Table	2.	(Contd.)
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Com- pound	¹ Η NMR, δ, ppm	IR, v, cm^{-1}
XIV	4.80 s, 4.95 s (2H, CH ₂ =C), 1.80 s (3H, CH ₃), 2.90 s (2H, CH ₂), 4.15 t (2H, CH ₂ O), 3.45 d (2H, CH ₂ Cl), 3.55 d (2H, OCH ₂), 3.90 m (1H, CHO) 3.10 br s (1H, OH)	730, 885, 905, 1165, 1660, 2245, 3010, 3380
XV	4.85 s, 4.70 s (2H, CH ₂ =C), 1.70 s (3H, CH ₃), 2.75 s (2H, CH ₂), 1.36 s [6H, (CH ₃) ₂], 3.40, 3.55 m (4H, CH ₂ Cl, CH ₂), 3.65 m (1H, OCH), 3.15s (1H, OH)	726, 890, 990, 1170, 1665, 2230, 3100, 3400
XVI	4.86–5.38 m (2H, CH ₂ =C), 5.57–6.05 m (1H, C=CH), 2.70–3.05 m (2H, CH ₂), 1.38 s [6H, (CH ₃) ₂], 3.65–3.80 m (2H, OCH ₂), 2.85–3.00 m (1H, CHO), 2.30–2.65 m (2H, CH ₂)	880, 910, 950, 1180, 1248, 1660, 2235, 3010, 3065
XVII	4.80 s, 4.95 s (2H, $CH_2=C$), 1.86 s (3H, CH_3), 2.90 s (2H, OCH_2), 4.20 t (2H, CH_2O), 3.50 m (2H, CH_2), 3.05 m (1H, OCH), 2.45–2.70 m (2H, OCH_2)	885, 915, 950, 990, 1170, 1255, 1640, 2225, 3010, 3065
XVIII	4.70 s, 4.90 s (2H, $CH_2=C$), 1.70 s (3H, CH_3), 2.86 s (2H, CH_2), 1.30 s [6H, $(CH_3)_2$], 3.45 m (2H, CH_2), 2.90 m (1H, OCH), 2.30–2.65 m (2H, OCH ₃)	890, 910, 953, 1167, 1250, 1645, 2210, 3060, 3100
XIX	4.75 s, 4.86 s (2H, CH ₂ =C), 1.76 s (3H, CH ₃), 2.88 s (2H, CH ₂), 4.10 t (2H, CH ₂ O), 3.20–3.60 m (1H, CH, 2H, CH ₂), 2.33–2.65 q [6H, N(CH ₂) ₂], 1.36 s [6H, (CH ₂) ₂]	886, 990, 1180, 1650, 2230, 2785, 3010, 3400
XX	4.70 s, 4.95 s (2H, CH ₂ =C), 1.70 s (3H, CH ₃), 2.85 s (2H, CH ₂), 1.35 s [6H, (CH ₃) ₂], 3.25–3.65 m (3H, CH, CH ₂), 2.30–2.60 q [6H, N(CH ₂) ₂], 1.30 s [6H, (CH ₃) ₂]	890, 985, 1160, 1655, 2240, 2786, 3100, 3450
XXI	4.73 s, 4.92 s (2H, CH ₂ =C), 1.80 s (3H, CH ₃), 2.30 s (2H, CH ₂), 1.35 s [6H, (CH ₃) ₂], 3.30–3.65 m (2H, OCH ₂), 2.80–3.00 m (1H, CH ₀), 2.40–2.70 m (2H, CH ₂ O), 1.38 s [6H, (CH ₃) ₂]	880, 915, 1045, 1175, 1650, 2230, 3100
XXII	4.80–5.40 m (2H, CH ₂ =C), 5.55–6.00 m (1H, C=CH), 2.75–2.90 m (2H, CH ₂), 1.32 s [6H, (CH ₃) ₂], 3.25–3.60 m (2H, OCH ₂), 2.95–3.15 m (1H, CHO), 2.30–2.65 m (2H, CH ₂ O), 1.36 s [6H, (CH ₃) ₂]	885, 990, 1050, 1150, 1640, 2235, 3010

Note: The data for compounds I-IV, which are not included in the table, are given in [10].

layer). The ¹H NMR spectra were recorded on a Tesla BS-487 B spectrometer (80 MHz) in carbon tetrachloride solutions using HMDS internal reference. The purity of the resulting compounds was controlled by thin-layer chromatography using Silufol UV-254 plates in various solvent mixtures and by gas–liquid chromatography on an LKhM-8 MD-5 device.

The physicochemical properties and spectra of compounds I-XXII are listed in Tables 1 and 2.

To prepare primary and tertiary allylacetylenic alcohols I–VI, dimethylformamide (100 ml), K_2CO_3 (1.5 g), CuI (9.5 g), and triethylamine (0.5 ml) were placed in a three-necked flask and stirred for 15– 20 min in a nitrogen flow at 50°C. Appropriate acetylene compound (propargyl alcohol or dimethylethynylcarbinol) (0.05 mol) was added, the reaction mixture was stirred for 2 h, and then appropriate alkenyl halide (allyl chloride, 2,3-dichloropropene, methallyl chloride) (0.1 mol) was added dropwise. The resulting mixture was stirred for an additional 8 h at $50-55^{\circ}$ C. After cooling, the mixture was washed with water and extracted with ether. The ether layer was separated, dried with MgSO₄, and evaporated in a vacuum. The yield of products **I–VI** was 65–80%. The physicochemical properties of the resulting compounds were close to those of the compounds prepared by the published procedure [10].

1-Acetoxy-5-hexen-2-yne VII. Acetic anhydride (4.1 g, 0.04 mol) was added with stirring to a mixture of allylacetylenic alcohol I (2 g, 0.02 mol) and concentrated sulfuric acid (0.1 ml) at $6-9^{\circ}$ C, and the reaction mixture was left overnight. Then, the mixture was stirred for 1.5 h on heating to $65-70^{\circ}$ C. After cooling, the reaction mixture was diluted with water, neutralized with K₂CO₃, and extracted with ether. The ether layer was dried (MgSO₄) and evaporated in a vacuum to yield compound **VII**.

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Compounds VIII–X were prepared by similar procedures using alcohols III, V, and VI.

2-Methyl-7-oxa-8-ethoxy-1-nonen-4-yne XI. Hydrochloric acid (33%, 0.3 ml) was added with stirring at 11°C to a mixture of allylacetylenic alcohol **III** (0.06 mol) and vinyl ethyl ether (0.06 mol). The flask was intermittently shaken; in so doing, the temperature of the reaction mixture increased to 42°C. The reaction mixture was left overnight. Then, the reaction mixture was neutralized and extracted with ether. The ether layer was dried (K₂CO₃), the solvent was evaporated in a vacuum, and compound **XI** was isolated by vacuum sublimation.

Acetal XII was prepared by a similar procedure using alcohol IV.

3-Chloro-1-(1,1-dimethyl-5-hexen-2-ynyloxy)propan-2-ol XIII. Epichlorohydrin (4 g, 0.043 mol) was added with stirring at $0-5^{\circ}$ C to allylacetylenic alcohol **II** (12.4 g, 0.1 mol) containing boron trifluoride etherate (0.1 ml). The reaction mixture was stirred for 4 h at 25°C, and compound **XIII** was isolated by vacuum distillation.

Chlorohydrins **XIV** and **XV** were prepared by similar procedures from alcohols **III** and **IV**.

1-(1,1-Dimethyl-5-hexen-2-ynyloxy)-2,3-epoxypropane XVI. Powdered KOH (8.4 g, 0.15 mol) was added with stirring at 8–10°C to a solution of chlorohydrin XIII (10.8 g, 0.05 mol) in ether (60 ml). The reaction mixture was stirred for 3 h at 12–14°C. After common work-up and removal of the solvent, compound XVI was isolated by vacuum distillation.

Epoxides **XVII**, **XVIII** were prepared similarly from chlorohydrins **XIV** and **XV**.

1-(5-Methyl-5-hexen-2-ynyloxy)-3-diethylaminopropan-2-ol XIX. A mixture containing epoxide XVII (3 g, 0.018 mol) and diethylamine (6.5 g, 0.5 mol) was stirred in the presence of water (2 ml)

Compound	Inhibitor concentration, g l ⁻¹	Corros	sion rate, g m⁻	² h ⁻¹	Degree of corrosion protection, %			
		40	60	80	40	60	80	
I	0.5	5.7	11.4	8.4	86.2	93.3	98.7	
	1.0	3.1	9.1	5.3	92.5	94.6	99.2	
Ш	0.5	5.7	46.5	11.4	86.3	72.7	98.3	
	1.0	3.8	24.3	9.7	90.8	85.7	98.5	
V	0.5	6.3	33.4	17.5	84.8	80.4	97.4	
	1.0	4.2	21.7	8.6	90.1	87.2	98.7	
VII	0.5	5.3	13.9	10.4	95.4	91.0	98.4	
	1.0	1.9	5.4	5.5	91.0	96.8	99.2	
VIII	0.5	3.3	13.0	5.3	92.6	92.4	99.2	
	1.0	2.4	4.7	1.1	96.2	97.4	98.8	
XI	0.5	0.3	1.1	1.3	99.2	99.3	99.8	
	1.0	0.2	0.6	0.6	99.4	99.7	99.9	
XII	0.5	1.35	3.3	21.0	96.7	98.0	98.0	
	1.0	2.0	2.6	3.8	95.1	98.4	99.7	
XIX	0.5	1.34	6.2	3.8	96.8	95.8	93.1	
	1.0	2.2	3.6	2.4	93.9	97.9	96.7	
XX	0.5	3.8	3.3	7.9	93.1	97.9	98.8	
	1.0	2.4	2.1	4.5	96.7	98.9	99.6	
XXI	0.5	4.36	10.3	14.0	88.9	94.0	97.2	
	1.0	2.60	5.0	10.6	94.6	96.0	98.4	

Table 3. Allylacetylene inhibitors of steel corrosion in 5 M H_2SO_4

for 8 h at 50°C, then dioxane (about 10 ml) was added to obtain the homogeneous mixture; in so doing, the temperature of the reaction mixture increased. The mixture was heated at 40°C for 3 h. Then it was extracted with ether and the ether layer was dried (K_2CO_3). The solvent was removed in a vacuum and compound **XIX** was isolated by vacuum distillation.

Amino alcohol **XX** was prepared similarly from epoxide **XVIII**.

2,2-Dimetyl-4-(3,3,7-trimethyl-2-oxo-7-octen-4-ynyl)-1,3-dioxolane XXI. Epoxide **XVI** (1.8 g, 0.01 mol) was added to a mixture of acetone (8,1 g, 0.14 mol) containing boron trifluoride etherate (1.8 g, 0.01 mol); in so doing, the temperature of the reaction mixture increased. After storage for 24 h, the reaction mixture was treated with a saturated aqueous solution of K_2CO_3 , and the organic layer was separated and dried (MgSO₄). The solvent was removed in a vacuum and 1,3-dioxolane **XXI** was isolated by vacuum distillation.

1,3-Dioxolane **XXII** was prepared similarly from epoxide **XVIII**.

Among all the allyl derivatives prepared, ten compounds (namely, **I**, **III**, **V**, **VII**, **VIII**, **XI**, **XII**, **XIX**– **XXI**) were studied as inhibitors of the acid corrosion of steel in 5 N H₂SO₄. The inhibition effect of compounds was studied by gravimetric procedure simulating the conditions of acidic etching of steel and rolled metal. Before experiments, the specimens $(30 \times 20 \times 3 \text{ mm})$ were cleaned on a polishing device with rubber discs, degreased with acetone, and stored for 1 day in a desiccator. The inhibitor concentration was $0.5-1.0 \text{ g l}^{-1}$; the required temperature of the acid medium of 40, 60, or 80° C was maintained with a thermostat. The experimental results listed in Table 3 show that all the above compounds are highly effective inhibitors of steel corrosion in sulfuric acid.

Along with studying the protective properties of compounds, we also tried to evaluate the correlation between the structure of organic compounds and their inhibiting performance and to determine the role of various functional groups in the total protection effect.

As seen, all the allylacetylenes studied exhibit pronounced activity in inhibition of the acid corrosion of steel. The degree of steel corrosion protection Z is 84-99%.

The compounds with normal structure with a terminal alcohol group I, III, and V are highly effective inhibitors. Substitution of the hydrogen atoms with chlorine atom and methyl groups slightly decreases the inhibiting performance of compounds V and III as

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compared to **I**. Allylacetylenes **VII** and **VIII** with a terminal acetoxy group are also effective inhibitors. Addition of the methylene group at the double bond enhances the inhibition properties at 40 and 60°C, but Z remains almost unchanged at 80° C.

Substitution of two hydrogen atoms with methyl groups (acetals **XI**, **XII**) slightly decreases the inhibiting performance of allylacetylenes. The protective effectiveness of amino alcohols **XIX** and **XX** is ambiguous.

On the whole, our experimental results suggest that functionalized allylacetylenes are highly effective inhibitors of the acid corrosion of steel; with increasing concentration of inhibitors their protection effect increases.

The performance of inhibitors increases with increasing temperature of the corrosion medium (especially at 80°C), which is probably due to the activation of chemisorption of the particles and molecular fragments on the metal surface. On substitution of methyl groups for hydrogen atoms, the structure of inhibitors becomes more branched, which hinders their adsorption on the metal surface and thus ordering in the resulting protective film. We assumed that the protection effect of allylacetylenes as inhibitors of the acid corrosion of steel is due to the π -electron interaction of the double and triple bonds with metal, resulting in the formation of chemisorption bonds at elevated temperatures. The contribution of the other fragments to corrosion inhibition is not decisive, because the overall inhibition effect is extremely high.

CONCLUSIONS

(1) Methods were developed for preparing primary and tertiary allylacetylenic alcohols in high yields by the reaction of alkenyl halides with the corresponding monosubstituted acetylenic alcohols in the presence of copper(I) iodide, triethylamine, and potassium carbonate in dimethylformamide.

(2) A series of functionalized allylacetylenes prepared are highly effective inhibitors of steel corrosion in 5 N sulfuric acid. The inhibition effect is primarily determined by the presence of double and triple bonds and is virtually independent of the nature of functional groups.

(3) With increasing temperature of the reaction mixture and concentration of the inhibitor, the protection effect increases.

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