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Lepidagathis alopecuroides methanol extract as corrosion inhibitor for mild steel in HCl

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Abstract. We report on the preliminary phytochemical screening of the methanolic extract of the aerial parts of *Lepidagathis alopecuroides* and its evaluation as a potential corrosion inhibitor for mild steel in aerated 1.0 M HCl by weight loss and linear polarization measurements. Weight loss measurements were conducted at 303, 313, 323 and 333 K. The results showed that *L. alopecuroides* inhibited the corrosion of mild steel in the acid solution with inhibition efficiency increasing with increase in the concentrations of the plant extract but decreased with increase in temperature. Linear polarization plots showed the plant extract to inhibit both the dissolution of the steel at the anode and the hydrogen evolution reaction (HER) at the cathode making it a mixed inhibitor. Temkin adsorption isotherms best modeled the adsorption of *L. alopecuroides* extract on the steel surface. From the values of the evaluated kinetic activation parameters in the study, the mechanism of physisorption is proposed for the adsorption of the plant extract on the steel surface.

Keywords: mild steel; corrosion inhibitor; linear polarization; activation energy; adsorption isotherm.

1. Introduction

Steel is a significant form of alloy because of its low cost and extensive engineering applications. Corrosion of steel structures leads to the malfunction and eventual breakdown of components and systems both in the processing and manufacturing industries, and diminishing life times of many components [1, 2]. The application of corrosion inhibitor formulations on steel structures is an effective and practical approach in mitigating the corrosion of steel parts exposed to different kinds of electrolytes [1, 2]. These formulations usually contain chemical compounds that possess hetero-atoms like oxygen, nitrogen and sulfur in aromatic or conjugated systems which serve as adsorption centers to the steel surface [3-7].

Many inorganic and organic compounds are reportedly in use as corrosion inhibitors against the corrosion of steel in aqueous media [8-10]. Most of these compounds are however toxic and are sources of environmental and health concerns. Hence, there is the need to search for environmentally friendly substitutes [3-7].

Plant materials are promising green corrosion inhibitors for steel as revealed by several studies [11-15]. Plant materials are not only organic but eco-friendly and pose less health concerns in respect of their toxic effects to man and animals compared to inorganic corrosion inhibitors like the salts of vanadium and chromium, and some toxic synthetic organic compounds [16]. Plant materials are rich sources of heterocyclic compounds like tannins, saponins, flavonoids and alkaloids [11, 12] which also contain heteroatoms.

The current study presents the preliminary phytochemical screening of the methanol extract of the aerial parts of *L. alopecuroides* plant and investigations of its potential as a corrosion inhibitor for mild steel in 1.0 M HCl.

2. Experimental

2.1. Materials
Mild steel strips were purchased from the steel market in Markurdi Nigeria. The chemicals of high purity (C_2H_5OH, CH_3COCH_3, $Pb(CH_3COO)_2$, NaOH, $CHCl_3$, $CH_3COOCOCH_3$, H_2SO_4, $FeCl_3$, CH_3COOH, NH_3, HCl, Wagner and Mayers' reagents) were obtained from Zayo-Sigma (ZSA) Chemicals Ltd - Jos. Nigeria and used without further purification.

2.2. Preparation of mild steel coupons
Mild steel strips were cut into the dimension of 30 x 20 x1.5 mm with holes of about 2.0 mm radius drilled into them. They were then polished with emery clothes, washed in ethanol, degreased in acetone, air-dried and stored in a desiccator prior to immersion in the test solutions for the experiments.

2.3. Preparation of plant extracts
Fresh aerial parts of *L. alopecuroides* were harvested from Emouha, Rivers state in Nigeria. *L. alopecuroides* plant materials were air dried and pulverized after which 300 g of the dried plant materials in 1 L of methanol were subjected to microwave assisted extraction [4, 17]. Plant extract was concentrated at 40 °C using a rotary evaporator (Heidolph Laborata 4002) [17] to obtain 12.72 g of the dark green extract having colloidal texture.

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2.4. Phytochemical screening

Preliminary phytochemical investigations were carried out using standard procedures as described by Ndukwe *et al.* [17].

Test for alkaloids. About 0.5 g of *L. alopecuroides* extract was stirred into 3 mL of 1% aqueous HCl on a steam bath and the mixture was filtered. 1 mL each of the filtrate was treated with 3 drops of Wagner's and Mayer's reagents, respectively [17]. Precipitation was observed for both reagents.

Test for flavonoids. (*i*) Lead acetate test. A 100 mg of *L. alopecuroides* extract was dissolved in 5 mL of distilled water, and $Pb(CH_3COO)_2$ solution was added to the solution [17]. Neither color change nor precipitation was observed. (*ii*) Sodium hydroxide test. 5 mL of 20% of NaOH was added to equal volume of dissolved *L. alopecuroides* extract (100 mg of the extract in 5 mL of distilled water) [17]. There was no observable change.

Test for steroids (Salkowski's test). *L. alopecuroides* extract (100 mg) was dissolved in 2 mL of chloroform. H_2SO_4 was carefully added to form a lower layer [17]. A reddish-brown color at the interface was observed.

Test for terpenes (Lieberman's test). About 0.5 g of *L. alopecuroides* extract was dissolved in 5 mL of chloroform, 1 mL of acetic anhydride was added, and then 1 mL of concentrated sulfuric acid was poured down the wall of the test tube to form a layer underneath [17]. There was formation of a reddish violet color at the junction of the two liquids and a green color in the chloroform layer.

Test for glycosides (Keller Killian test). *L. alopecuroides* extract (100 mg) was dissolved in 1 mL of glacial acetic acid containing 1 drop of $FeCl_3$ solution. This was then under laid with 1 mL concentrated H_2SO_4 [17]. A brown ring was observed at the interface.

Test for tannins. About 0.5 g of *L. alopecuroides* extract was stirred into 1 mL of distilled water, filtered and $FeCl_3$ solution added to the filtrate [17]. There was no observable reaction. Therefore, no blue-green precipitate was observed.

Test for saponins (Frothing test). A 0.5 g of *L. alopecuroides* extract was shaken with distilled water in a test tube. This was warmed on a water bath [17]. There was frothing or foaming which persisted after warming.

Test for anthraquinones (Borntrager's test). About 0.5 g of *L. alopecuroides* extract was taken into a dry test tube and 5 mL of chloroform was added and shaken for 5 min. The solution was filtered, and the filtrate shaken with an equal volume of 25% NH_3 solution [17]. There was no change observed, thus, no observable bright pink color in the upper aqueous layer.

2.5. Preparation of *L. alopecuroides* solution as corrosion inhibitor

Test solutions for gravimetric measurements were prepared as reported in earlier studies by Chahul *et al.* [6, 18, 19]. Various concentrations of 0.2 g/L, 0.4 g/L, 0.6 g/L, 0.8 g/L, and 1.0 g/L of *L. alopecuroides* extract were prepared in 100 mL of 1.0 M HCl solution and used for the experiments. A blank was prepared without the plant extract to serve as a control.

2.6. Weight loss measurements

Weight loss measurements were done as described elsewhere [12, 19, 20]. Already cleaned and pre-weighed mild steel (MS) coupons were immersed in duplicates in 100 mL of 1.0 M solution of HCl in the absence and presence of different concentrations of *L. alopecuroides* extract and left for the period of 3, 6, 9, 12, 24, 48, 72, 96 and 120 h. The coupons were removed from the test solutions each time, washed in water, cleaned with ethanol, dried with acetone, and reweighed. The effect of temperature on the corrosion of mild steel in the acid medium in the absence and presence of various concentrations of *L. alopecuroides* was investigated in a thermostated water bath at 303, 313, 323 and 333 K at 2 h immersion period.

The weight loss was taken to be the disparity between the initial and final weights of the mild steel coupons at a given time.

The difference in weights ($g·h^{-1}$) was calculated using Equation 1 [12, 20]:

$$\Delta w = w_2 - w_1 \qquad (1)$$

where $\Delta w = w_2 - w_1$ is the weight loss of metal after a given time. Inhibition efficiency (%IE) and surface coverage (θ) were calculated using:

$$IE_{exp} = \left(1 - \frac{w_{inh}}{w_{blank}}\right) \times 100 \qquad (2)$$

$$\theta = 1 - \frac{w_{inh}}{w_{blank}} \qquad (3)$$

where w_{inh} and w_{blank} are the weight loss (g) of the mild steel in the presence and absence of the inhibitor respectively [12, 20].

2.7. Linear polarisation analysis

A Metrohm Autolab AUT50280 potentiostat with a three-electrode design that has an MS working electrode, Pt auxiliary electrode and saturated calomel electrode (SCE) as reference electrode was employed to perform linear polarisation analysis. Mild steel coupons of 1 x 1 cm^2 dimensions, sealed with epoxy resin in such a way that only 1 cm^2 surface area left uncovered, were employed for the experiment. Linear polarization was conducted at room temperature in the potential range of -1500 to 1500 mV and a scan rate of 0.012 v/s.

The anodic and cathodic Tafel curves were extrapolated to corrosion potential to obtain the corrosion current densities (I_{corr}) [14]. Inhibition efficiency was calculated using Equation 4 [5, 20]:

$$IE\ (\%) = \frac{I_{corr(blank)} - I_{corr(inh)}}{I_{corr(blank)}} \times 100 \qquad (4)$$

where $I_{corr(blank)}$ is the corrosion current density of the steel coupon in the acid solution in the absence of *L. alopecuroides* and $I_{corr(inh)}$ is the corrosion current density of the steel coupon in the presence of *L. alopecuroides* [5, 20].

3. Results and discussion

3.1. Phytochemical screening

Results of the preliminary phytochemical screening of methanol extract of the aerial parts of *L. alopecuroides*

show the presence of alkaloids, saponins, steroids, terpenes and glycosides but flavonoids, tannins and anthraquinones were absent.

3.2. Weight loss studies

The effect of immersion time on weight loss of the steel coupons was investigated as illustrated in Figure 1. The plots in Figure 1 depict a remarkable steady decrease in the weight loss of the metal coupon as *L. alopecuroides* concentration increased. From this result, it can be inferred that *L. alopecuroides* extract retarded the corrosion of the steel coupons thereby inhibiting their corrosion in the understudied acid medium. This is corroborated by the increase in inhibition efficiency as observed in Figure 2. Equations 2 and 3 relates the proportionality of *IE* and *θ* meaning as *IE* increased with increasing *L. alopecuroides* concentration, the surface of the steel covered by *L. alopecuroides* extract also increased thereby making the steel surface inaccessible by the aggressive species in the acid medium, hence retarding its dissolution.
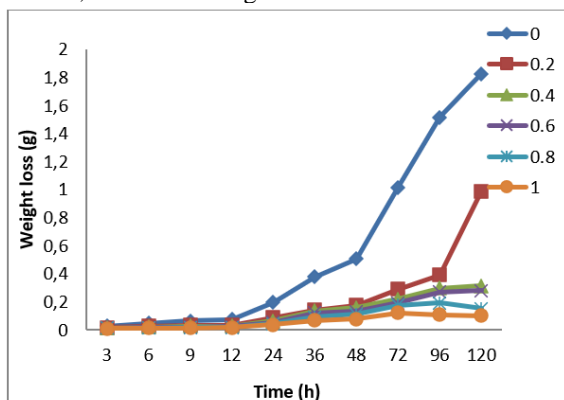

Figure 1. Variation of weight loss with time for the corrosion of mild steel in 1.0 M HCl in the absence and presence of different concentrations of *L. alopecuroides* at 303 K.
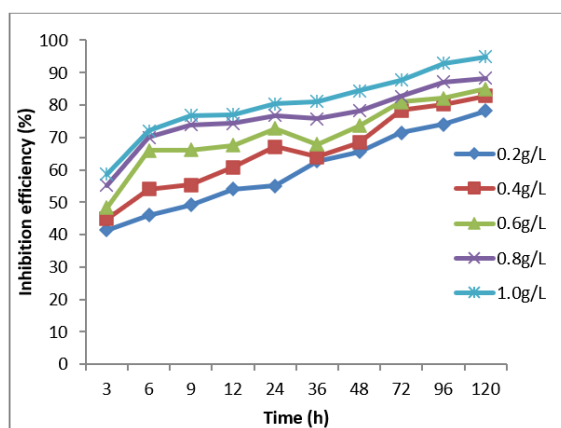

Figure 2. Variation of *IE* with time for the corrosion of mild steel in 1.0 M HCl in the presence of various concentrations of *L. alopecuroides* at 303 K.

Figure 3 shows a decrease in *IE* with increase in temperature indicating that at higher temperatures, there was less adsorption of *L. alopecuroides* components to the surface of the metal. This condition typifies physical adsorption mechanism as corroborated by other findings [8, 20-23]. The decrease in *IE* with increase in temperature means that the adsorbed components of *L. alopecuroides* on the steel surface were electrostatically

bond to the steel surface and at as the temperature of the system increased, there was increase in the kinetic motion of the molecules in the corrodent and subsequent agitation of the adsorbed species on the steel surface causing their desorption from it with a resulting decrease in surface coverage of the inhibitor and *IE*.
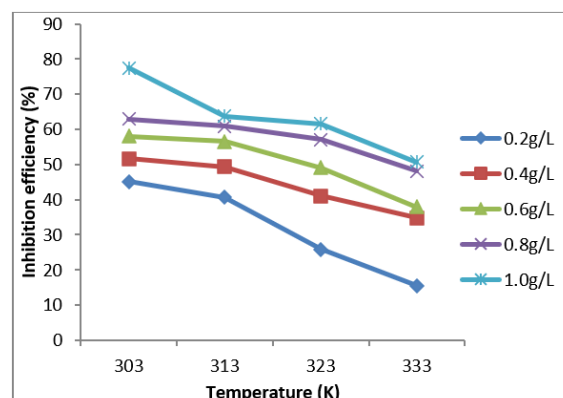

Figure 3. Variation of *IE* with temperature for the corrosion of mild steel in 1.0 M HCl in the presence of different concentrations of *L. alopecuroides* at 303 K.

3.3. Linear polarization

The relationship between the current and potential for the corrosion of mild steel electrode in the inhibited and uninhibited test solutions is illustrated in the Tafel plots in Figure 4. The presence of *L. alopecuroides* in the test solution can be observed to affect both the anodic and cathodic half reactions, moving the corrosion potential (E_{corr}) toward a more positive value and decreasing the anodic and cathodic current densities and the corresponding corrosion current density (i_{corr}). This means that *L. alopecuroides* is a mixed-type inhibitor in the corrosion inhibition of mild steel in the HCl medium [5, 7, 20, 21]. The inhibition efficiency of *L. alopecuroides* extract was calculated using Equation 4 and presented on Table 1.
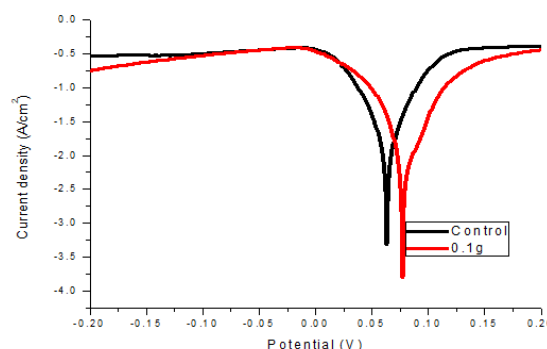

Figure 4. Tafel plots for the corrosion of mild steel in 1.0 M HCl in the presence of *L. alopecuroides*.

Table 1. Linear polarization parameters for the corrosion of mild steel in 1.0 M HCl in the presence of *L. alopecuroides*.

Conc (g/L)	E_{corr} (V)	I_{corr} (A/cm²)	βa (V/dec)	βc (V/dec)	CR (g/h)	IE (%)
Blank	0.063	0.824	0.313	0.405	0.429	-
0.1	0.076	0.116	0.130	0.214	0.060	85.92

3.4. Temperature and kinetics

The Arrhenius equation describes the relationship between the temperature of the system and the corrosion rate of the steel coupon [20-23]:

$$CR = Aexp\left(-\frac{E_a}{RT}\right) \qquad (5)$$

where *CR* is the corrosion rate of the steel coupon in $g \cdot cm^{-2} \cdot h^{-1}$, E_a is the activation energy of the corrosion and inhibition process, *A* is the pre-exponential factor, *T* is the absolute temperature (K) and *R* is the universal gas constant.

Taking the logarithm of both sides of Equation 5 yields,

$$-logCR = \log A - \frac{Ea}{2.303RT} \qquad (6)$$

The plot of *logCR vs.* 1/T for the corrosion of mild steel in the absence and presence of different amounts of *L. alopecuroides* extract is illustrated in Figure 5a. The activation energies are presented on Table 2.
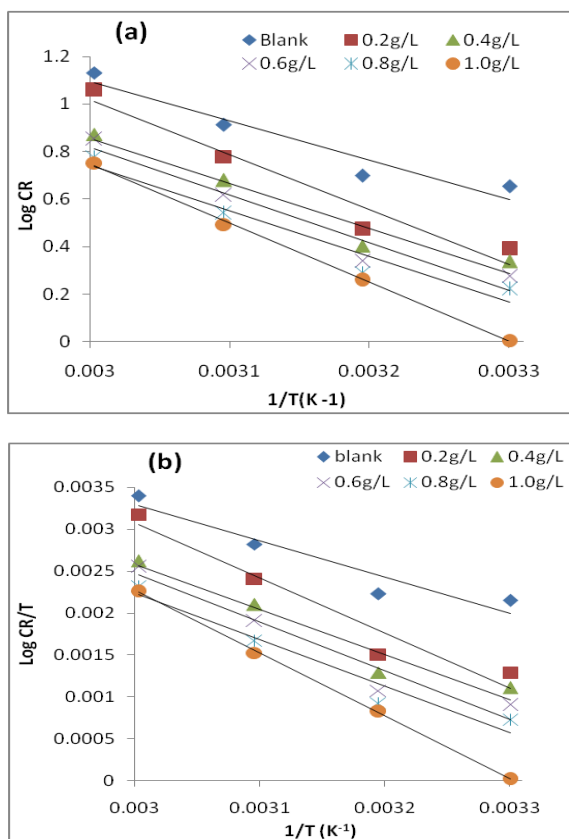

Figure 5. (a) Arrhenius and (b) Transition state plots for the corrosion of mild steel in the absence and presence of different levels of *L. alopecuroides* in 1.0 M HCl.

The activation energies for the corrosion of mild steel in the absence and presence of different amounts of *L. alopecuroides* plant extract in the acid medium are presented on Table 2.

The value of E_a in the uninhibited system not only increased on addition of *L. alopecuroides* extracts, but all the values of E_a in both the inhibited and free acid systems were less than 80 kJ/mol signifying a physisorptive kind of adsorption mechanism as earlier suggested.

The other activation parameters (ΔS^* and ΔH^*) for the adsorption of *L. alopecuroides* extracts on the steel coupon were calculated using Equation 7 [6, 20-23],

$$\log\left(\frac{CR}{T}\right) = \left[log\left(\frac{R}{Nh}\right) + \frac{\Delta S^*}{2.303R}\right] - \frac{\Delta H^*}{2.303RT} \qquad (7)$$

where ΔS^* is change in entropy of activation, ΔH^* is the change in enthalpy of activation, *CR* is the corrosion rate at absolute temperature *T*, *h* is the Planck's constant, *N* is the Avogadro's constant, (6.02E-23) and *R* is the universal gas constant.

A plot of log *CR/T* versus *1/T* is a straight-line graph with a slope of $\left(-\frac{\Delta H*}{2.303R}\right)$ and an intercept of $\left[log\left(\frac{R}{nh}\right) + \frac{\Delta S*}{2.303R}\right]$ (Figure 5b) from which the values of ΔH^* and ΔS^* were calculated respectively and presented on Table 2 [21-23].

Table 2. Values of E_a, ΔH^* and ΔS^* for the corrosion of mild steel in 1.0 M HCl in the absence and presence of different concentrations of *L. alopecuroides* extract.

Test	E_a (kJ/mol)	ΔH^* (kJ/mol)	ΔS^* (kJ/mol)
Blank	30.872	82.92	-197.28
0.2 g/L	42.969	126.06	-197.16
0.4 g/L	34.495	102.65	-197.24
0.6 g/L	37.105	110.92	-197.22
0.8 g/L	35.544	106.01	-197.24
1.0 g/L	48.047	143.35	-197.12

The negative entropies of activation suggest the association mechanism of the corrosion process whereby a decrease in disorder takes place ongoing from the reactants to the activated complex as reported in other studies [12, 22, 23].

3.5. Adsorption studies

The adsorption properties of *L. alopecuroides* extract were analyzed by fitting the obtained data for surface coverages at 303, 313, 323, and 333 K into different adsorption isotherm models. The Temkin adsorption isotherm gave the best fit. Equation 8 is the mathematical (linear form) form Temkin adsorption isotherm,

$$-2 \propto \theta = 2.303(logK_{ads} + logC) \qquad (8)$$

where *C* is the concentration of *L. alopecuroides* in the bulk electrolyte, θ is the degree of surface coverage of *L. alopecuroides*, K_{ads} is the adsorption equilibrium constant and $\propto$ is the molecular interaction parameter.

By plotting *logC* against θ at the understudied temperatures as shown in Figure 6, the R^2 values show that the adsorption pattern fits more perfectly into this model as the values were close to unity.
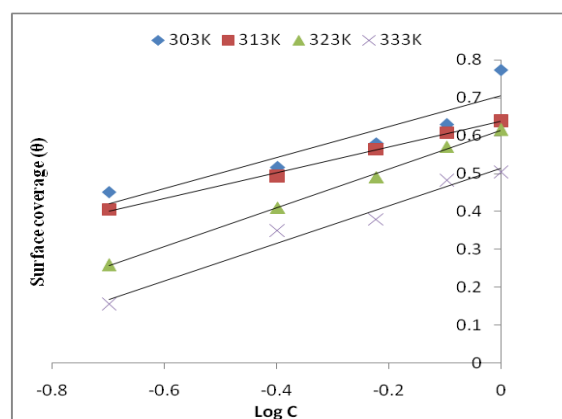

Figure 6. A plot of Temkin adsorption isotherm for the adsorption of *L. alopecuroides* extraction the surface of mid steel in 1.0 M HCl.

The free energy of adsorption, $\Delta G^o{}_{ads}$, can be calculated using Equation 9:

$$\Delta G^o{}_{ads} = -2.303 RT \log(55.5 K_{ads}) \qquad (9)$$

Values of adsorption parameters deduced from the plots are presented on Table 3.

Table 3. Temkin isotherm parameters for the adsorption of *L. alopecuroides* extract on the surface of mild steel in 1.0 M HCl at different temperatures.

Temperature	K_{ads}	$\Delta G^o{}_{ads}$ (kJ/mol)	R^2
303 K	2.026	-11.897	0.8508
313 K	1.892	-11.724	0.9958
323 K	1.849	-11.666	0.9982
333 K	1.671	-11.411	0.9729

The values of K_{ads} in Table 3 are low which is indicative of interactions that are not strong between the plant extract and the surface of the steel coupon. Higher values of K_{ads} have been reported to illustrate greater adsorption of the adsorbate (plant extract) on the adsorbent (steel surface), better inhibition efficiency and vice versa [8, 25]. This supports physisorptive mechanism of adsorption of *L. alopecuroides* extract on the steel surface [8, 25].

The negative values of $\Delta G^o{}_{ads}$ obtained on Table 3 infer that the adsorption of *L. alopecuroides* extract on the steel surface was spontaneous [21-24]. Values of $\Delta G^o{}_{ads} < -40$ kJ/mol is linked with electrostatic interactions between inhibitor molecules and the charged metal/alloy surface (physisorption) [21-24]. The values of $\Delta G^o{}_{ads}$ obtained in this study were < -20 kJ/mol (Table 3) confirming physical adsorption of the *L. alopecuroides* extract on the steel surface.

4. Conclusion

This study has shown that *L. alopecuroides* contains alkaloids, saponins, steroids, terpenes and glycosides, and served as an excellent inhibitor for the corrosion of mild steel in 1.0 M HCl. Inhibition efficiency of the plant extract increased with increase in concentration of the extract but decreased with increase in temperature. Tafels plots revealed *L. alopecuroides* inhibited both the dissolution of the steel at the anode and the hydrogen evolution reaction at the cathode making it a mixed inhibitor. Evaluated activation energy, E_a and $\Delta G^o{}_{ads}$ values reveal the corrosion inhibition process to be physisorptive and spontaneous while the Temkin isotherm model described the adsorption mechanism of *L. alopecuroides* to the steel surface.

Conflict of interest

The authors do not declare any conflict of interest.

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