### **Original Research Article**

# Inhibition of Mild Steel Corrosion in Acidic Medium by *Telfairia occidentalis* Rind Extract

### ABSTRACT

*Telfairia occidentalis* rind extract has been studied as a potential green inhibitor for mild steel corrosion in 1 M  $H_2SO_4$  using weight loss and hydrogen evolution methods. The results of the investigation reveal that *Telfairia occidentalis* rind extract is a good inhibitor of mild steel corrosion in sulphuric acid solution. The inhibition efficiency increases with increase in rind extract concentration but decrease with increase in temperature. The calculated thermodynamic parameters reveal that the corrosion inhibition process was endothermic and spontaneous. Physical adsorption has been proposed for the adsorption of *Telfairia occidentalis* rind extract onto mild steel surface. The adsorption of the extract on mild steel surface obeys the Langmuir adsorption isotherm.

Keywords: Telfairia occidentalis, mild steel, corrosion inhibition, physisorption, Langmuir isotherm

#### 12 13 **1. INTRODUCTION**

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15 Corrosion of metals is a global phenomenon that has received enormous attention and concern, due 16 to its degrading effects on metallic structures and infrastructure. Mild steel, which is widely used in 17 industries, is often exposed to aggressive environments during industrial processes such as picking, 18 descaling and acid cleaning, which results in its corrosion. The cost of replacing corroded metallic 19 equipment is usually very high. In order to protect metals in aggressive environments, the use of 20 corrosion inhibitors is practiced globally [1]. Corrosion inhibitors are compounds containing nitrogen, 21 oxygen, sulphur and/or phosphorus [2]. Though many synthetic organic and inorganic compounds 22 have been reported to exhibit good anti-corrosive property [3-5], their usage has been discouraged 23 due to their toxicity, which makes them environmentally unfriendly. Thus safety and environmental 24 concern have prompted researchers to find eco-friendly corrosion inhibitors from natural products 25 such as plant extracts containing phytochemicals like alkaloids, saponins, terpenes, tannins, etc. 26 which are rich in oxygen, sulphur and nitrogen [6]. Besides being eco-friendly, plant extracts are 27 renewable, readily available, cheap and biodegradable. Some of the plant extracts which have been 28 investigated by researchers as showing good anti-corrosive property on mild steel in acidic medium 29 include; Stachytarpheta indica leaf extract [7], Phoenix dactylifera seed extract [8], watermelon rind 30 extract [9], Piper longum fruit extract [10], Fenugreek seed extract [11], and Gmelina arborea stem 31 bark extract [12].

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32 Telfairia occidentalis commonly called fluted pumpkin in English, lkong-ubong by the Efik/Ibibio and 33 ugu by the Igbo ethnic groups in Nigeria belongs to the family Cucurbitaceae. The climbing vegetable 34 has edible leaves borne on tender shoots. The shoots also bear large gourd fruits containing edible 35 seeds [13]. The seeds are used for cultivation. The seeds are housed by a big gourd which is used as 36 feed for herbivores such as pigs and goats. The shoot (stem) and leaves have been found to be rich 37 in antioxidants and have antimicrobial properties [14]. The leaf and shoot are also used in herbal 38 medicine by the people of Nigeria. No work has been reported on the anti-corrosive property of 39 Telfairia occidentalis rind extract on mild steel. The aim of this work was to investigate the corrosion 40 inhibition effect of *Telfairia occidentalis* rind extract on mild steel corrosion in H<sub>2</sub>SO<sub>4</sub> solution.

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### 2. MATERIAL AND METHODS

### 44 **2.1 Test materials**

Mild steel sheet used for this work was obtained from Kenjohnson Metals, Uyo, Nigeria. It had the following chemical composition (weight %): C (0.12), Mn (0.85), S(0.06), P (0.05), Si (0.09) and Fe (98.83). The sheet was mechanically press - cut into 4 cm x 4 cm coupons for the weight loss method and 2 cm x 4 cm for the hydrogen evolution studies. Mill scales on these coupons were removed by polishing to mirror finish using different grades of silicon carbide papers. The coupons were degreased in absolute ethanol, dipped in acetone before air-drying. They were then stored in moisture – free desiccator before use in corrosion studies.

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### 55 **2.2 Preparation of** *Telfairia occidentalis* rind extract

57 Fresh rinds of Telfairia occidentalis were collected from Itam Market in Uyo, Akwa Ibom State, 58 Nigeria. They were washed and oven – dried at 50  $^{\circ}$ C until constant weight. They were then ground to 59 powder. As reported in literature [15], the dried ground sample of Telfairia occidentalis rind was 60 macerated with 90% ethanol for seven days, with intermittent stirring, at room temperature in glass 61 trough with cover. The mixture was then filtered. The filtrate was evaporated at 40  $^{\circ}$ C in a water bath 62 to constant weight, leaving a brownish rind extract in the beaker. Extract concentrations of 0.5 g/L, 1.0 63 g/L, 1.5 g/L and 2.0 g/L respectively were prepared using 1 M H<sub>2</sub>SO<sub>4</sub> solution for both the weight loss 64 and hydrogen evolution studies.

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### 67 2.3 Phytochemical screening of *Telfairia occidentalis* rind extract

Phytochemical screening of *Telfairia occidentalis* ethanol rind extract was performed using standard
 procedures reported in literature [16 - 17].

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#### 71 2.4 Weight Loss Method

Previously weighed mild steel coupons were suspended with the aid of glass hooks and rods and immersed in 100 ml of 1 M H<sub>2</sub>SO<sub>4</sub> solution (blank) and in 1 M H<sub>2</sub>SO<sub>4</sub> solution containing 1.0 g/L – 2.0 g/L *Telfairia occidentalis* rind extract (inhibitor), respectively, in open beakers. In each experiment, one mild steel coupon per beaker was used. The beakers were then placed in a thermostatic water bath maintained at 30 °C, 40 °C, 50 °C, and 60 °C, respectively. The mild steel coupons were retrieved from the test solutions after four (4) hours and scrubbed with bristle brush under running water. They were dipped in acetone and air - dried before reweighing.

79 The inhibition efficiency I(%) was calculated using the formula [18]: 80

$$I(\%) = \left(1 - \frac{W_1}{W_0}\right) \times 100\tag{1}$$

where  $W_0$  and  $W_1$  are the weight losses of the mild steel coupons in the absence and presence of extract, respectively, in 1 M H<sub>2</sub>SO<sub>4</sub> at the same temperature.

85 The corrosion rate (CR) was calculated using the equation:

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$$CR (mg cm-2 hr-1) = \left(\frac{W}{At}\right)$$
(2)

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89 where W is the weight loss (mg), A is the total surface area  $(cm^2)$  while t is the exposure time (hr).

#### 90 2.5 Hydrogen Evolution Method

The reaction vessel and procedure for measuring the corrosion process by this method are as described by Abakedi and Asuquo [6]. Two mild steel coupons weighing 8.0 g were dropped into 100 ml of 1 M  $H_2SO_4$  solution (blank). The volume of  $H_2$  gas evolved from the corrosion reaction was recorded every 60 seconds for 60 minutes. The same experiment was repeated in the presence of 0.5 g/L - 2.0 g/L *Telfairia occidentalis* rind extract (inhibitor) in 1 M  $H_2SO_4$  solution.

96 The hydrogen evolution rate  $R_H$  was calculated using the equation:

$$R_{\rm H} \,({\rm cm}^3{\rm min}^{-1}) = \left(\frac{V_1 - V_0}{t_1 - t_0}\right) \tag{3}$$

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100 where  $v_1$  and  $v_0$  are the volumes of hydrogen gas evolved at time  $t_1$  and  $t_0$  respectively.

101 The inhibition efficiency by the hydrogen evolution method was obtained through the formula [19]:

$$I(\%) = \left(1 - \frac{R_{\rm Hi}}{R_{\rm H0}}\right) \times 100 \tag{4}$$

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where  $RH_0$  and  $RH_i$  are the rates of evolution of  $H_2$  gas in the absence and presence of extract, respectively, at a specified time.

### 108 3. RESULTS AND DISCUSSION

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### 110 3.1 Results of Phytochemical Screening of *Telfairia occidentalis* Rind Extract

111 The results of the phytochemical screening of ethanol *Telfairia occidentalis* rind extract revealed the

112 presence of tannins, flavonoids, saponins, anthraquinones and phlobatannins.

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114 3.2 Effect of *Telfairia occidentalis* Rind Extract Concentration on Inhibition Efficiency 115 The variation of the inhibition efficiency with concentration of rind extract in 1 M H<sub>2</sub>SO<sub>4</sub> solution is shown in Fig.1. At a particular temperature, it is observed that the inhibition efficiency increased with 116 117 increase in rind extract concentration. The maximum inhibition efficiency of the rind extract was 118 89.00% at extract concentration of 2.0 g/L at 30°C. The effect of Telfairia occidentalis rind extract 119 (TORE) on the volume of H<sub>2</sub> gas evolved with time for the mild steel corrosion in 1 M H<sub>2</sub>SO<sub>4</sub> at  $30^{\circ}$ C 120 are shown in Fig. 2. It is observed that increase in the rind extract concentrations led to a decrease in 121 the volume of H<sub>2</sub> gas evolved at a particular time. Table 1 reveals that the calculated values of 122 inhibition efficiency in the presence of TORE increased with increase in extract concentration. The 123 maximum inhibition efficiency was 90.94% at extract concentration of 2.0 g/L. The inhibition 124 efficiencies obtained from both the weight loss and hydrogen evolution methods followed a similar 125 trend. An increase in inhibition efficiency with increase in inhibitor concentration indicates a strong 126 interaction between the mild steel surface and the inhibitor [3]. The corrosion inhibiting effect of the 127 extract can be attributed to the phytochemical constituents of the extract. However, the inhibiting

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- 128 effect of the adsorption of inhibitors on the metal surface may be difficult to attribute to any particular
- 129 constituent, since most of these constituents are known to inhibit corrosion [20].
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133Fig. 1. Variation of inhibition efficiency (%) with Telfairia occidentalis rind extract134concentration (g/L) for mild steel corrosion in 1 M H2SO4 at different temperatures135

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Table 1. Effect of *Telfairia occidentalis* rind extract concentration on inhibition efficiency of
 mild steel in 1 M H₂SO₄ solution at 30 °C (Hydrogen evolution measurements)

Extract Concentration	H <sub>2</sub> Evolution Rate	Inhibition Efficiency
(g/L)	(cm <sup>3</sup> /min)	(%)
Blank	0.4967	-
0.5	0.1983	60.07
1.0	0.1083	78.19
1.5	0.0717	85.57
2.0	0.0450	90.94

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Fig. 2. Variation of volume of H<sub>2</sub> gas evolved (cm<sup>3</sup>) with time (min) for mild steel corrosion in 1 M H<sub>2</sub>SO<sub>4</sub> in the absence and presence of *Telfairia occidentalis* rind extract at 30 °C

### 148 **3.3 Effect of Temperature on inhibition Efficiency**

149 The effect of temperature on the inhibition effect of Telfairia occidentalis rind extract on mild steel 150 corrosion in 1 M  $H_2SO_4$  solution are shown Table 2. It is observed that an increase in temperature 151 resulted in a decrease in the inhibition efficiency. The decrease in inhibition efficiency indicates that 152 the extract was more effective in inhibiting mild steel corrosion at lower temperatures than at higher 153 temperatures. Furthermore, a decrease in inhibition efficiency with increase in temperature signifies 154 physical adsorption of the extract on mild steel surface. The adsorption of an inhibitor at the metal 155 solution interface involves the displacement of water molecules adsorbed at the metal surface [21]: 156  $lnh_{(sol)} + nH_2O_{(ads)} \rightarrow lnh_{(ads)} + nH_2O_{(sol)}$ (5) The inhibitor can combine with freshly generated Fe<sup>2+</sup> ions on the steel surface to form metal-inhibitor 157 158 complexes:  $Fe \rightarrow Fe^{2+} + 2e^{-}$ 159 (6)  $Fe^{2+} + inh \rightarrow (Fe-Inh)_{ads}$ 160 (7)161 An increase in the temperature of the system can result in instability of the Fe-Inh complex leading to

- 162 poor inhibition of corrosion at higher temperatures, if the complex is soluble at high temperature.
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### 167 Table 2. Weight loss data for mild steel corrosion in 1 M $H_2SO_4$ in the absence and presence of 168 different concentrations of *Telfairia occidentalis* rind extract at 30 °C – 60 °C

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Extract	Corrosion rate			Inhibition efficiency				
Concentration	$(mg cm^{-2} hr^{-1})$			(%)				
	30°C	40°C	50°C	60°C	30°C	40°C	50°C	60°C
Blank	2.5102	5.2078	9.0203	20.8336	-	-	_	-
0.5 g/L	0.9141	2.3008	4.4156	10.9258	63.59	55.82	51.05	47.56
1.0 g/L	0.5555	1.2367	2.7063	7.2313	77.87	76.25	69.99	65.29
1.5 g/L	0.4133	0.9945	2.0883	5.4992	83.54	80.90	79.85	73.60
2.0 g/L	0.2758	0.7266	1.5328	4.3109	89.01	86.05	83.01	79.31

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### 172 . 3.4 Thermodynamic Consideration

173 The alternative formulation of Arrhenius equation was used to obtain the activation energy  $(E_a)$ 

values for mild steel corrosion in 1 M  $H_2SO_4$  in the presence and absence of TORE [17]:

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$$\ln CR = \frac{-E_a}{RT} + \ln A \tag{8}$$

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where CR is the corrosion rate, R is the universal gas constant, T is the temperature in Kelvin while A
is the pre-exponential factor.

181 Plots of In CR against 1/T for mild steel corrosion in 1 M H<sub>2</sub>SO<sub>4</sub> in the absence and presence of 182 different concentrations of TORE are presented in Fig. 3. The values of the activation energies  $(E_a)$ 183 were obtained from the gradients of the plots and are presented in Table 3. Table 3 reveals that the 184  $E_a$  values in the presence of the extract were higher than the  $E_a$  value of the blank (57.355 kJ/mol), 185 indicating an increase in the energy barrier of the reaction in the presence of the TORE when 186 compared to the blank. These higher activation energies imply slower reaction rate for the corrosion of 187 mild steel in H<sub>2</sub>SO<sub>4</sub> solutions in the presence of the extract than in its absence. The higher E<sub>a</sub> values 188 in the H<sub>2</sub>SO<sub>4</sub>-TORE medium signifies physical adsorption [22]. It can therefore be proposed that 189 Telfairia occidentalis rind extract physically adsorbed onto the mild steel surface.



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Fig. 3. Plot of In CR against 1/T (Arrhenius plot) for mild steel corrosion in 1 M H<sub>2</sub>SO<sub>4</sub> in the
 absence and presence of *Telfairia occidentalis* rind extract

196 In order to establish the mode of adsorption of the inhibitors on the mild steel surface, the values of 197 enthalpy of activation ( $\Delta H^{o}_{ads}$ ) and entropy of activation ( $\Delta S^{o}_{ads}$ ) were obtained from an alternative 198 formulation of the transition state equation [6]:

$$\ln\left(\frac{CR}{T}\right) = \left[\ln\left(\frac{R}{Nh}\right) + \frac{\Delta S_{ads}^{\circ}}{R}\right] - \frac{\Delta H_{ads}^{\circ}}{RT}$$
(8)

200 201 where CR is the corrosion rate, T is temperature in Kelvin, R is the universal gas constant, N is 202 Avogadro's number, A the Arrhenius pre-exponential factor, while h is the Planck's constant. Fig 4 203 indicates the plots of In (CR/T) against 1/T for mild steel corrosion in 1 M H<sub>2</sub>SO<sub>4</sub> in the absence and 204 presence of different concentrations of TORE. The values of  $\Delta H^{o}_{ads}$  were obtained from slopes and 205 intercepts of the plots and are presented in Table 3. The values of  $\Delta H^{o}_{ads}$  in the blank and in the 206 presence of the extract were positive. The positive values of  $\Delta H^{o}_{ads}$  both in the blank and in the 207 presence of extracts signify the endothermic nature of the mild steel corrosion process. It has been 208 explained by some workers [23] that since in an endothermic reaction the molecules absorb heat from 209 the surrounding, therefore increasing the number of molecules (by increasing the extract 210 concentration) leads to an increase in the amount of heat absorbed. Hence, the observed increase in 211 the values of  $\Delta H^{o}_{ads}$  as extract concentration increases. The negative values of  $\Delta S^{o}_{ads}$  indicate a



212 decrease in the disorderliness of the adsorption process of TORE on mild steel surface.

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Fig.4. Plot of In (CR/T) against 1/T (Transition state plot) for mild steel corrosion in 1 M H<sub>2</sub>SO<sub>4</sub> in the absence and presence of *Telfairia occidentalis* rind extract

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Table 3. Calculated values of thermodynamic parameters for mild steel corrosion in 1 M  $H_2SO_4$  in the absence and presence of *Telfairia occidentalis* rind extract

Extract	Ea	ΔH	ΔS
Concentration	(kJ mol <sup>-1</sup> )	(kJ mol <sup>-1</sup> )	$(JK^{-1} mol^{-1})$
Blank	57.355	54.711	-56.888
0.5 g/L	67.305	64.664	-32.161
1.0 g/L	70.524	67.885	-26.216
1.5 g/L	70.728	68.088	-27.746
2.0 g/L	76.449	72.142	-17.618

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### 225 **3.6 Adsorption Studies**

Several adsorption isotherms such as the Langmuir, Temkin, Freundlich and El-awady isotherms were
tested using the weight loss measurements. The best fit for the adsorption of TORE on mild steel
surfaces were found to obey the Langmuir adsorption isotherm given as [24]:

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \tag{10}$$

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where  $\theta$  is the degree of surface coverage, C is the concentration of inhibitor and K<sub>ads</sub> is the equilibrium constant of the adsorption process. Fig. 5 reveals linear plots of C/ $\theta$  against C with gradient of 'n' and 1/K<sub>ads</sub> as intercepts. Values of K<sub>ads</sub> (Table 4) were found to decrease with increase

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in temperature, indicating that TORE became loosely adsorbed onto the mild steel surface withincrease in temperature.



Fig.5. Plot of C/ $\theta$  against C (Langmuir isotherm) for mild steel corrosion in 1 M H<sub>2</sub>SO<sub>4</sub> in the absence and presence of *Telfairia occidentalis* rind extract

245 The standard free energy of adsorption ( $\Delta G^{o}_{ads}$ ) was calculated using equation (11) [3]:

$$K_{ads} = \frac{1}{55.5} \exp\left(\frac{-\Delta G_{ads}^{\circ}}{RT}\right)$$
(11)

where R is the universal gas constant, 55.5 is the molar concentration of water in the solution, T is

248 the temperature in Kelvin while K<sub>ads</sub> is the equilibrium constant of adsorption.

### Table 4. Some parameters of the linear regression of Langmuir adsorption isotherm for mild steel corrosion in 1 M H<sub>2</sub>SO<sub>4</sub> containing *Telfairia occidentalis* rind extract

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Temperature	R <sup>2</sup>	n	$I/K_{ads}(g/L)$	K <sub>ads</sub> (g⁻¹L)	ΔG° <sub>ads</sub> (kJ mol⁻¹)	
303K	0.9990	0.98	0.3050	3.2787	-13.110	
313K	0.9977	0.97	0.3893	2.5687	-12.908	
323K	0.9991	0.96	0.4891	2.0446	-12.707	
333K	0.9999	0.98	0.5563	1.7976	-12.744	

The negative values of  $\Delta G^{\circ}_{ads}$  shown in Table 4 indicate that mild steel corrosion inhibition by TORE occurred spontaneously. Values of  $\Delta G^{\circ}_{ads}$  less negative than -20 kJ mol<sup>-1</sup> indicate physical adsorption while values of  $\Delta G^{\circ}_{ads}$  more negative than -40 kJ mol<sup>-1</sup> are attributed to chemical adsorption of inhibitor onto the metal surface [25 – 26]. Since the values of  $\Delta G^{\circ}_{ads}$  obtained in this study are less negative than -20 kJ mol<sup>-1</sup>, physical adsorption of the extract onto mild steel surface has been proposed. This is further supported by a decrease in the inhibition efficiency with increase in temperature.

### 270 **3.7 Scanning Electron Microscopy (SEM) Analysis**

SEM analysis was carried out to investigate the surface morphology of mild steel coupons immersed in 1 M  $H_2SO_4$  solution in the absence and presence of 2.0 g/L *Telfairia occidentalis* rind extract (TORE) for four (4) hours at 30 °C. Fig. 5(a) reveals a damage mild steel surface due to severe corrosion of mild steel in the blank. Conversely, Fig. 5(b) reveals that mild steel surface corroded less in the presence of the extract relative to the blank, as reflected in a smoother surface. This indicates that the extract protected the mild steel surface by adsorbing on it.

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Fig. 5. SEM image of mild steel surface immersed for 4 hours in (a) 1 M H<sub>2</sub>SO<sub>4</sub> solution (Blank) and (b) 1 M H<sub>2</sub>SO<sub>4</sub> solution containing 2.0 g/L *Telfairia occidentalis* rind extract

### 282 4. CONCLUSION

The following conclusions can be drawn from this work: *Telfairia occidentalis* rind extract is a good inhibitor for mild steel in  $H_2SO_4$  solution at  $30 \,^{\circ}C - 60 \,^{\circ}C$ . The inhibition efficiency increased with increase in extract concentration but decreased with increase in temperature. The higher values of activation energy (E<sub>a</sub>) in the extract relative to the blank coupled with a decrease in inhibition efficiency with increase in temperature in addition to the  $\Delta G^{\circ}_{ads}$  of the process being less negative than -20 kJ mol<sup>-1</sup>, physical adsorption has been proposed for the adsorption of the extract onto mild steel surface. The positive values of  $\Delta H^{\circ}_{ads}$  and the negative values of  $\Delta G^{\circ}_{ads}$ , respectively, indicate

291 the endothermic and spontaneous nature of adsorption of the extract on mild steel surface. The

adsorption of the extract on mild steel surface obeyed the Langmuir adsorption isotherm.

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