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Corrosion inhibition and statistical data of thiocarbanilide and thiocarbanilide admixed with 4-hydroxy-3-methoxybenzaldehyde on carbon steel in dilute acid media

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Abstract. The inhibition performance of thiocarbanilide (TD) and thiocarbanilide admixed with 4-hydroxy-3-methoxybenzaldehyde (TDHM) on the corrosion of mild steel in 1M H_2SO_4 and HCl acid solutions was studied through coupon measurement. Data obtained showed the optimal performance of TD is 71.69% in H_2SO_4 and 83.14% HCl acid solution respectively. TDHM performed more effectively with optimal inhibition efficiency of 96.77% and 97.21% in both acids. The result showed the performance of corrosion inhibiting compounds improves through synergistic interaction with other compounds. However, exposure time and inhibitor concentration strongly influenced the performance of the compounds at varying degrees. Statistical data shows the standard deviation for inhibition efficiency of the compounds was significantly higher in H_2SO_4 compared to HCl due to the high degree of variation analogous to exposure time and stability of the inhibitor molecules. TD and TDHM generally showed lower standard of deviation, higher mean value, lower margin of error and higher proportion of inhibition efficiency data above 70% inhibition performance. Statistical data from analysis of variance depict both exposure time and inhibitor concentration is statistically relevant, and influences the inhibition performance of TD and TDHM compounds. However, the influence of concentration is significantly limited compared to exposure time with statistical relevance values above 60%

1. Introduction

Carbon steels are the most universally applicable and produced steel worldwide, with a production volume of about 85% of the total annual global steel manufactured. It is the largest category of ferrous metals and alloys in weight and total price [1]. Corrosion of carbon steels has been a major problem for industrial establishments and governmental organizations due to its enormous economic consequence. It is the most prevalent cause of accidents and downtime in process industries. In some industrial applications, appropriate selection of corrosion resistant materials for construction is possible. However, such method is not cost effective [2]. The limited corrosion resistance of carbon steels is responsible for its short lifespan during service and continuous replacement when damaged [3]. There are other effect techniques of corrosion control. However, despite their relative effectiveness and high cost of operation, they do not provide the universal protection afforded by corrosion inhibitors. Ongoing research and practical field applications has resulted in the formulation of effective corrosion inhibitors for numerous applications from the application in vehicular radiators transport to desalination plants, mining, energy generation, chemical production and petrochemical refinery [4-7]. Corrosion inhibitors are intermittently added to corrosive environments where they attach onto the steel or modify the corrosive environment [8-10]. Most of the effective corrosion inhibitors in industry are toxic and increasingly being subjected to government regulations on usage and application [11-13]. Research on less toxic compounds of organic origin has produced promising results, thus the need for further test [14-16]. In contribution to the research on organic corrosion inhibitors, this study investigates the electrochemical and corrosion

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inhibition property of thiocarbanilide and thiocarbanilide admixed with 4-hydroxy-3-methoxybenzaldehyde compound on mild steel in HCl and H_2SO_4 solution.

2. Experimental methods

Thiocarbanilide (TD) was oncocted in cubic concentratess of 0%, 0.0025%, 0.005%, 0.0075%, 0.01%, 0.0125% and 0.015%. Thiocarbanilide admixed with 4-hydroxy-3-methoxybenzaldehyde (TDHM) in equal proportions was concocted in cubic concentrates of 0%, 0.13%, 0.25%, 0.38%, 0.50%, 0.63% and 0.75% respectively. The inhibitor concentrations were formulated in 200ml of 1M of H_2SO_4 and 1M HCl acid solution. Carbon steel (MS) rods were machined into 14 test specimens with general configuration of 0.7 cm radius and 0.7 cm length. The surface ends of the steel specimens were abraded with emery papers of different grits. Weight measures of MS specimens were separately immersed in 200 ml of the H_2SO_4 and HCl solution at the formulated TD and TDHM concentrations for 240 h. The prepared MS were weighed at 24 h hiatus with Ohaus analytical weighing balance. Tabulated results of MS weight-loss at specific TD and TDHM concentrations in the acid media are shown from Table 1 to 4. The weight loss is the variation between the first weight of the steel (maintained for 168 h) and the final weight obtained at 24 h interval. Tables 5-8 shows the data of inhibition efficiency (*IE*) calculated from the equation below;

$$IE = \left[\frac{W_1 - W_2}{W_1}\right] * 100 \quad (1)$$

W_1 and W_2 are the weight-loss of the control and inhibited MS in the acid media with respect to exposure time.

3. Results and discussion

3.1 Coupon measurement

Tables 1-4 shows the data for weight loss of MS in TD/ H_2SO_4, TD/HCl, TDHM/ H_2SO_4 and TD/HCl solution for 240 h. The presence of TD and TDHM inhibitor compound in the acid media significantly reduced the weight-loss of the MS specimens. However, TD inhibition effect in H_2SO_4 tends to be time dependent with the weight loss results of the inhibited MS steel decreasing gradually till 240 h. The same phenomenon occurred for MS in TD/HCl solution. However, the weight-loss values are slightly lower due to effective inhibition action of TD in HCl. TDHM performed more effectively in H_2SO_4 and HCl solution compared to TD. TDHM significantly decreased the weight-loss of MS in both acids. Tables 5 to 8 shows the data for inhibition efficiency of TD and TDHM in H_2SO_4 and HCl solution. Effective inhibition action of the compounds was observed at 240 h of exposure. Observation of Table 5 shows TD inhibition performance varies slightly with respect to concentration. Secondly, after the lowest TD concentration exposure time strongly influenced the performance of TD i.e. the performance of TD improved with exposure time due to the slow molecular action of the inhibitor species. The corresponding data of TD inhibition on MS in HCl solution shows TD performed more effectively from the onset of the exposure hour with minimal variation in inhibition efficiency compared to TD in H_2SO_4 solution. The performance of TDHM on MS in H_2SO_4 and HCl solution were generally similar with gradual change in value from the onset of the exposure hours. Fig. 1(a) and Fig. 1(b) show the plots of TD inhibition efficiency with exposure time at the highest and lowest inhibitor concentration in H_2SO_4 and HCl solution while Fig. 2(a) and (b) shows the corresponding plots for TDHM in H_2SO_4 and HCl solution. Observation of Fig. 1(a) shows the wide variation of TD plot configuration at highest and lowest TD concentration compared to the plots in Fig. 1(b). Secondly, the plot configuration at lowest TD concentration in Fig. 1(a) appears to be stable with respect to exposure time compared to the plot at highest TD concentration. The plots in Fig. 1(b) shows similar configuration with respect to exposure time.

Table 1. Data on weight-loss of MS from 1M H_2SO_4/TD (0% - 0.015%) solution

TD conc. (%) / Exp. Time(h)	0%	0.003%	0.005%	0.008%	0.010%	0.013%	0.015%
24	0.063	0.018	0.058	0.056	0.052	0.050	0.062
48	0.110	0.039	0.075	0.086	0.071	0.060	0.105
72	0.158	0.061	0.093	0.096	0.089	0.071	0.143
96	0.243	0.076	0.110	0.114	0.104	0.097	0.163
120	0.307	0.092	0.124	0.129	0.122	0.116	0.183
144	0.403	0.128	0.150	0.153	0.141	0.139	0.208
168	0.478	0.146	0.153	0.173	0.158	0.141	0.233
192	0.646	0.186	0.188	0.220	0.196	0.205	0.273
216	0.739	0.213	0.217	0.229	0.215	0.221	0.289
240	0.848	0.246	0.244	0.240	0.241	0.253	0.321

Table 2. Data on weight-loss of MS from 1M HCl/TD (0% - 0.015%) solution

TD conc. (%) / Exp. Time(h)	0%	0.003%	0.005%	0.008%	0.010%	0.013%	0.015%
24	0.076	0.034	0.028	0.034	0.042	0.018	0.034
48	0.134	0.042	0.041	0.043	0.074	0.037	0.046
72	0.191	0.050	0.055	0.053	0.098	0.056	0.058
96	0.288	0.063	0.077	0.060	0.126	0.068	0.068
120	0.406	0.075	0.091	0.071	0.136	0.081	0.079
144	0.645	0.099	0.115	0.094	0.182	0.108	0.099
168	0.851	0.119	0.136	0.115	0.211	0.131	0.118
192	1.372	0.232	0.218	0.203	0.281	0.230	0.197
216	1.616	0.288	0.264	0.251	0.321	0.280	0.252
240	1.852	0.346	0.320	0.312	0.363	0.323	0.317

Table 3. Data on weight-loss of MS from 1M H_2SO_4/TDHM (0% - 0.75%) solution

TD conc. (%) / Exp. Time(h)	0%	0.13%	0.25%	0.38%	0.50%	0.63%	0.75%
24	0.182	0.061	0.072	0.073	0.062	0.054	0.076
48	0.357	0.077	0.098	0.081	0.084	0.069	0.089
72	0.656	0.091	0.114	0.096	0.095	0.079	0.102
96	1.078	0.103	0.123	0.102	0.096	0.085	0.112
120	1.509	0.117	0.130	0.105	0.102	0.090	0.115
144	1.945	0.128	0.140	0.114	0.107	0.095	0.121
168	2.391	0.143	0.150	0.120	0.113	0.102	0.125
192	2.810	0.150	0.161	0.127	0.121	0.106	0.132
216	3.344	0.167	0.166	0.134	0.127	0.113	0.141
240	3.785	0.177	0.179	0.144	0.135	0.122	0.150

Table 4. Data on weight-loss of MS from 1M HCl/TDHM (0% - 0.75%) solution

TD conc. (%) / Exp. Time(h)	0%	0.13%	0.25%	0.38%	0.50%	0.63%	0.75%
24	0.266	0.030	0.040	0.042	0.095	0.024	0.036
48	0.558	0.047	0.059	0.063	0.118	0.039	0.054
72	0.975	0.067	0.078	0.081	0.137	0.058	0.070
96	1.623	0.081	0.093	0.099	0.155	0.067	0.081
120	2.281	0.089	0.103	0.115	0.167	0.079	0.093
144	2.958	0.108	0.121	0.133	0.179	0.087	0.107
168	3.629	0.120	0.134	0.148	0.203	0.101	0.116
192	4.087	0.134	0.146	0.167	0.217	0.114	0.126
216	4.315	0.148	0.162	0.184	0.241	0.122	0.140
240	4.835	0.159	0.174	0.205	0.265	0.142	0.152

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Table 5. Data on inhibition efficiency of TD in 1M H_2SO_4

Exp. Time(h) \ TD conc. (%)	0.003%	0.005%	0.008%	0.010%	0.013%	0.015%
24	71.25	8.37	10.90	17.22	21.64	2.53
48	64.22	31.79	21.83	35.78	45.65	4.53
72	61.36	41.24	38.90	43.27	55.27	9.14
96	68.57	54.70	53.25	57.21	59.97	32.95
120	69.90	59.52	57.89	60.27	62.30	40.48
144	68.16	62.89	62.05	64.93	65.52	48.35
168	69.42	68.06	63.88	67.02	70.51	51.17
192	71.20	70.91	65.96	69.68	68.32	57.68
216	71.11	70.64	69.01	70.88	70.07	60.94
240	71.02	71.27	71.69	71.63	70.22	62.15

Table 6. Data on inhibition efficiency of TD in 1M HCl

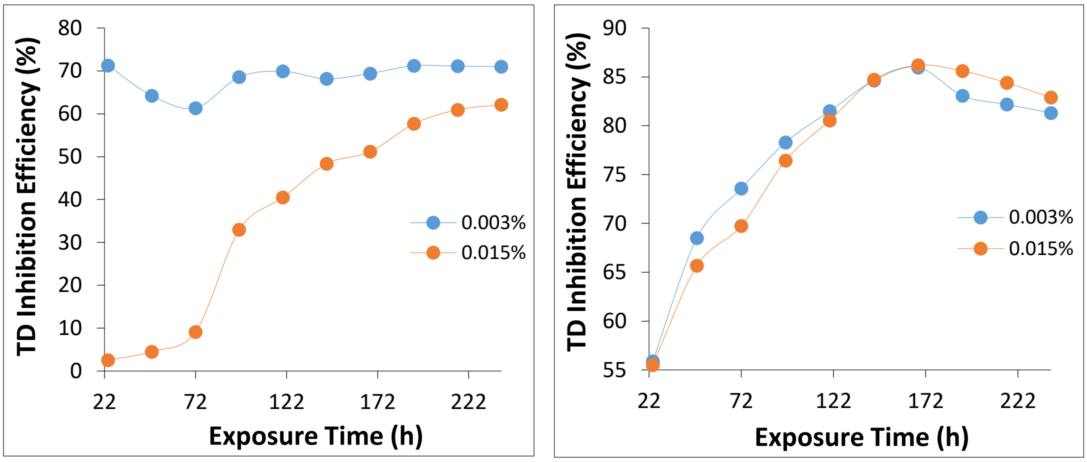
Exp. Time(h) \ TD conc. (%)	0.003%	0.005%	0.008%	0.010%	0.013%	0.015%
24	55.89	63.87	55.89	44.63	75.92	55.50
48	68.51	69.18	67.76	44.95	72.25	65.67
72	73.57	71.32	72.53	48.72	70.80	69.75
96	78.29	73.40	79.15	56.33	76.38	76.45
120	81.51	77.51	82.62	66.59	80.13	80.55
144	84.64	82.15	85.48	71.85	83.26	84.72
168	85.97	84.06	86.48	75.20	84.58	86.20
192	83.07	84.10	85.22	79.48	83.23	85.62
216	82.18	83.67	84.50	80.12	82.66	84.40
240	81.32	82.71	83.14	80.41	82.57	82.89

Table 7. Data on inhibition efficiency of TDHM in 1M H_2SO_4

Exp. Time(h) \ TD conc. (%)	0.13%	0.25%	0.38%	0.50%	0.63%	0.75%
24	66.47	60.70	59.93	65.81	70.47	58.23
48	78.32	72.55	77.31	76.39	80.62	75.13
72	86.18	82.66	85.42	85.54	87.95	84.40
96	90.49	88.61	90.58	91.09	92.16	89.66
120	92.23	91.38	93.03	93.25	94.03	92.39
144	93.41	92.78	94.12	94.49	95.14	93.78
168	94.03	93.73	95.00	95.26	95.74	94.77
192	94.65	94.26	95.49	95.71	96.24	95.31
216	95.02	95.02	95.98	96.21	96.62	95.78
240	95.33	95.27	96.19	96.44	96.77	96.05

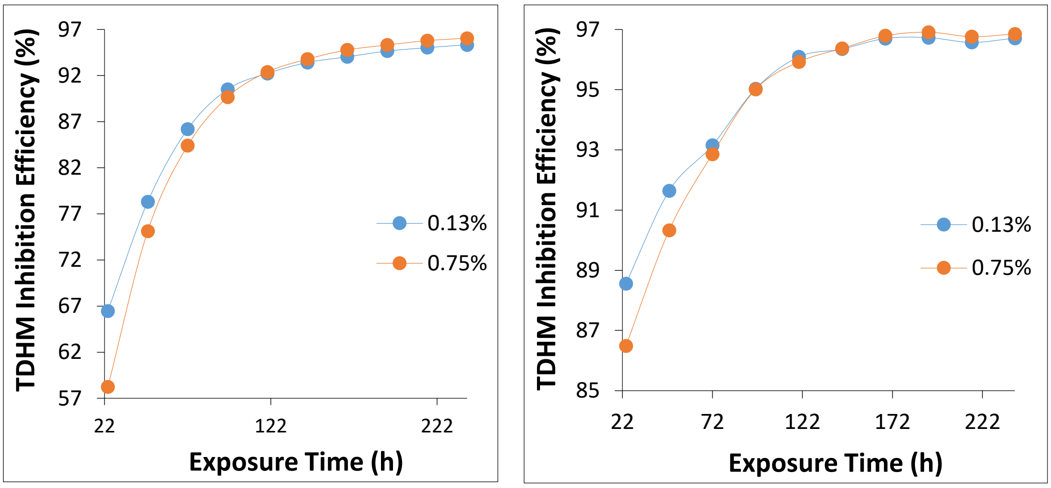
Table 8. Data on inhibition efficiency of TDHM in 1M HCl

Exp. Time(h) \ TD conc. (%)	0.13%	0.25%	0.38%	0.50%	0.63%	0.75%
24	88.56	85.06	84.09	64.45	91.16	86.49
48	91.64	89.49	88.67	78.80	93.10	90.33
72	93.15	91.98	91.73	85.97	94.03	92.86
96	95.03	94.26	93.91	90.42	95.85	95.01
120	96.09	95.46	94.94	92.70	96.55	95.92
144	96.35	95.91	95.51	93.95	97.05	96.37
168	96.70	96.31	95.91	94.41	97.21	96.79
192	96.73	96.42	95.91	94.68	97.21	96.91
216	96.57	96.24	95.75	94.41	97.17	96.76
240	96.71	96.40	95.76	94.52	97.06	96.85



(a) (b)

Figure 1. Plot of TD concentration versus exposure time at highest and lowest TD concentration (a) in H_2SO_4 solution and (b) in HCl solution



(a) (b)

Figure 2. Plot of TD concentration versus exposure time at highest and lowest TDHM concentration (a) in H_2SO_4 solution and (b) in HCl solution

3.2 Statistical evaluation

The mean, standard deviation and margin of error for TD and TDHM inhibition efficiency data in H_2SO_4 and HCl solution are shown in Table 9 and 10. Observation of Table 9 shows the standard deviation of TD inhibition efficiency data with respect to concentration are significantly higher in H_2SO_4 solution after the lowest TD concentration compared to the values in HCl solution due to the high degree of variation of the inhibition efficiency values from mean value i.e. variation of the values over time are significant and TD inhibition performance tends to be less stable in the H_2SO_4 solution. The higher the TD concentration, the lower the mean value in H_2SO_4, and the higher the standard deviation due high variation of inhibition efficiency data over time. This further confirmed from the average inhibition values in H_2SO_4 which are lower than the values obtained in HCl. The relatively lower standard deviation values in HCl solution shows TD inhibition performance varies slightly over time i.e. less time dependent. The margin of error shows that 22% and 77% of inhibition efficiency data obtained in H_2SO_4 and HCl solution are above 70% inhibition efficiency at margin of error of $\pm$10.42% and $\pm$10.70%. Observation of Table 10 shows the standard deviation for TDHM inhibition efficiency in H_2SO_4 are significantly higher than the values obtained in HCl solution due to the lower degree of variation of inhibition efficiency over time in HCl solution. This confirms TDHM inhibition performance in HCl is more stable and effective as shown in the mean inhibition value which are generally above 90%. However, the mean inhibition value of TDHM in H_2SO_4 is higher than the values obtained for TD in H_2SO_4 and HCl solution. The margin of error shows 92% and 100% of TDHM inhibition efficiency are above 70% inhibition value at margin of error of ±6.99% and ±0%.

Table 9. Statistical data for mean, standard deviation and margin of error for TD inhibition efficiency data in H_2SO_4 and HCl solution

H_2SO_4						
TD Concentration	0.003%	0.005%	0.008%	0.010%	0.013%	0.015%
Standard Deviation	3.33	20.84	20.87	18.10	15.31	23.59
Mean	68.62	53.94	51.54	55.79	58.95	36.99
Proportion above 70% Inhibition Efficiency	22%	Margin of Error	±10.42%			
HCl						
TD Concentration	0.003%	0.005%	0.008%	0.010%	0.013%	0.015%
Standard Deviation	9.25	7.32	9.97	14.86	4.98	10.35
Mean	77.50	77.20	78.28	64.83	79.18	77.18
Proportion above 70% Inhibition Efficiency	77%	Margin of Error	±10.70%			

Table 10. Statistical data for mean, standard deviation and margin of error for TDHM inhibition efficiency data in H_2SO_4 and HCl solution

H_2SO_4						
TDHM Concentration	0.13%	0.25%	0.38%	0.50%	0.63%	0.75%
Standard Deviation	9.39	11.57	11.61	10.29	8.69	12.22
Mean	88.61	86.70	88.31	89.02	90.57	87.55
Proportion above 70% Inhibition Efficiency	92%	Margin of Error	±6.99%			
HCl						
TDHM Concentration	0.13%	0.25%	0.38%	0.50%	0.63%	0.75%
Standard Deviation	2.79	3.82	3.98	9.86	2.14	3.52
Mean	94.75	93.75	93.22	88.43	95.64	94.43
Proportion above 70% Inhibition Efficiency	100%	Margin of Error	±0%			

Analysis of variance (ANOVA) was used to evaluate the statistical importance of inhibitor concentration and exposure time (sources of variation) on the protection efficiency of TD and TDHM in H_2SO_4 and HCl solution. The ANOVA data for TD and TDHM inhibition performance is shown in Tables 11 and 12. The statistical relevance factor presents the percentage significance of inhibitor concentration and exposure time. The mean square ratio represents the significance factor which must be greater than the theoretical significance factor to be statistically relevant. The statistical relevance factor in Table 11 shows exposure time is statistically more relevant on the inhibition performance of TD in H_2SO_4 and HCl solution with values of 62.31% and 66.12%. These values are greater than the statistical relevance value of inhibitor concentration with values of 22.96% and 21.37%. Observation of the value for mean square ratio shows they are greater than the theoretical significance factor, thus both exposure time and TD concentration influence the inhibition performance of TD compound though exposure time is statistically more significant. The statistical relevance factor in Table 12 shows exposure time overwhelmingly influence the inhibition performance of TDHM inhibitor in H_2SO_4 solution with value of 96.99% compared to the influence of TDHM concentration at 1.4%. However, the mean square ratio for both sources of variation is greater than the theoretical significance factor signifying both are statistically relevant and at varying degrees influence the performance of TDHM compound in H_2SO_4 solution. The ANOVA data for TDHM in HCl solution aligns with the values earlier discussed for TD compound in H_2SO_4 and HCl solution.

Table 11. ANOVA data for TD inhibition performance in H_2SO_4 and HCl solution

Source of Variation	**Addition of Squares**	**Degree of Freedom**	**Mean Square**	**Mean Square Ratio (F)**	**Theoretical Significance Factor**	**Statistical Relevance Factor, F (%)**
H_2SO_4						
TD Conc.	5362.396	5	1072.48	14.03	2.42	22.96
Exp. Time	14553.78	9	1617.09	21.15	2.15	62.31
Residual	3440.61	45	76.46			
Total	23356.79	59				
HCl						
Source of Variation	**Addition of Squares**	**Degree of Freedom**	**Mean Square**	**Mean Square Ratio (F)**	**Theoretical Significance Factor**	**Statistical Relevance Factor, F (%)**
TD Conc.	1445.76	5	289.15	15.37	2.42	21.37
Exp. Time	4473.35	9	497.04	26.41	2.15	66.12
Residual	846.85	45	18.82			
Total	6765.96	59				

Table 12. ANOVA data for TDHM inhibition performance in H_2SO_4 and HCl solution

Source of Variation	**Addition of Squares**	**Degree of Freedom**	**Mean Square**	**Mean Square Ratio (F)**	**Theoretical Significance Factor**	**Statistical Relevance Factor, F (%)**
H_2SO_4						
TDHM Conc.	87.69	5	17.54	7.78	2.42	1.40
Exp. Time	6084.68	9	676.08	299.78	2.15	96.99
Residual	101.49	45	2.26			
Total	6273.86	59				
HCl						
Source of Variation	**Addition of Squares**	**Degree of Freedom**	**Mean Square**	**Mean Square Ratio (F)**	**Theoretical Significance Factor**	**Statistical Relevance Factor, F (%)**
TDHM Conc.	327.46	5	65.49	8.32	2.42	19.28
Exp. Time	1016.63	9	112.96	14.35	2.15	59.87
Residual	354.13	45	7.87			
Total	1698.22	59				

4. Conclusion
Thiocarbanilide and thiocarbanilide admixed with 4-hydroxy-3-methoxybenzaldehyde effectively inhibited the corrosion of mild steel in H_2SO_4 and HCl solution with optimal inhibition efficiency above 70% and 80% for thiocarbanilide in H_2SO_4 and HCl while the corresponding performance for thiocarbanilide admixed with 4-hydroxy-3-methoxybenzaldehyde is above 90% in both acid media. The performance of both compounds varies at differing with respect to time and concentration. Statistical data shows only a small proportion of inhibition efficiency data of thiocarbanilide in H2SO4 are above 70% inhibition efficiency compared to the other values obtained in the acid media which is above 75%. Data from ANOVA shows exposure time strongly influences the performance of both compounds compared to inhibitor concentration

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