

Corrosion inhibition of mild steel in nitric acid media by some Schiff bases derived from anisalidine

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Corrosion inhibition performance of mild steel in nitric acid solution containing different concentration of anisalidine derivative Schiff bases viz. N- (4-nitro phenyl) p-anisalidine (SB1), N- (4-chloro phenyl) p-anisalidine (SB2), N- (4-phenyl) p-anisalidine (SB3), N- (4-methoxy phenyl) p-anisalidine (SB4), N- (4-hydroxy phenyl) p-anisalidine (SB5) has been investigated using mass loss, thermometric and potentiostatic polarization technique. Inhibition efficiencies of Schiff bases have been evaluated at different acid strength. The inhibition efficiency was found larger than their parent amines. Inhibition efficiencies of synthesized Schiff bases increase with inhibitor concentration. Inhibition efficiency increases up to 98.32% with anisalidine derivative Schiff base.

Keywords: Schiff base, degradation, inhibitor and mild steel.

INTRODUCTION

Mild steel is widely used as an engineering material and its corrosion in acidic medium is of great economic importance. Schiff bases have gained importance over the years because of their use as corrosion inhibition. The exposure can be most severe but in many cases, corrosion inhibitors are widely used in industry to prevent or to reduce corrosion rate of metallic materials in acidic media^{1,2}. Schiff bases possess $-C=N-$ imine linkage which is supposed to be responsible for their complexing behaviors towards metal ions and corrosion inhibition action^{3,4}. Corrosion commonly occurs at metal surfaces in the presence of oxygen and moisture, involving two electrochemical reactions. Oxidation takes place at the anodic site and reduction occurs at the cathodic site. In acidic medium hydrogen evolution reaction predominates. Corrosion inhibitors reduce or prevent these reactions. They are adsorbed on metal surface and form a barrier to oxygen and moisture by complexing with metal ions or by removing corrodants from the environment. Some of the inhibitors facilitate the formation of the passivating film on the metal surface.

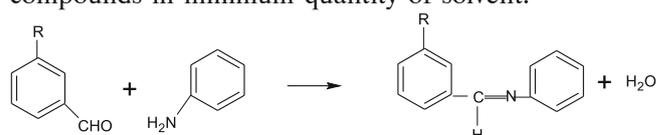
Generally, the organic compounds containing hetero atoms like O, N, S, and in some cases Se and P are found to have the function as very effective corrosion inhibitors⁵⁻¹⁴. The efficiency of these compounds depends upon electron density present around the heteroatoms¹⁵. Inhibition efficiency also depends upon the number of adsorption active centers in the molecule, their charge density, molecular size, mode of adsorption and formation of metallic complexes. Heteroatoms capable of forming coordinate-covalent bond with metal owing to their free electron pairs. Also the compounds with π -bonds generally exhibit good inhibitive properties due to an interaction of π -orbital with their structure which makes them effective potential corrosion inhibitors¹⁶.

Corrosion of mild steel and its alloys in different acid media has been extensively studied^{17,18}. The present research paper deals with the preparation of five Schiff bases viz. N- (4-nitro phenyl) p-anisalidine (SB1), N- (4-chloro phenyl) p-anisalidine (SB2), N- (4-phenyl) p-anisalidine (SB3), N- (4-methoxy phenyl) p-anisalidine (SB4), N- (4-hydroxy phenyl) p-anisalidine (SB5).

EXPERIMENTAL

Preparation of Schiff bases

Schiff bases are prepared by refluxing equimolar quantities of respective carbonyl compounds with amino compounds in minimum quantity of solvent.



Where R = H, alkyl or aryl.

Specimen preparation

Mild steel specimens of the composition of 0.12% C, 0.32% Mn, 0.05% S, 0.02% SiO₂ and the rest of Fe of rectangular specimens dimension 5.0*2.5*0.02 cm³ containing a small hole of 2 mm diameter near the upper edge were employed for the determination of corrosion rate in a beaker containing 50 ml of test solution at 23°C with a precision of $\pm 0.5^\circ\text{C}$ and left exposed to air. While being buffed to produce a mirror finish of mild steel, they were cleaned with emery paper then degreased with acetone. The specimens were suspended by glass hook; the degree of surface coverage (θ) can be calculated as¹⁹

$$\theta = \frac{(\Delta M_u - \Delta M_i)}{\Delta M_u}$$

Where θ surface coverage and ΔM_u and ΔM_i are the mass loss of the metal in uninhibited and inhibited acid. The corrosion rate mmpy (millimeter penetration per year) can be obtained by the following equation:

$$\text{Corrosion rate (mmpy)} = \frac{(543 \times W)}{(\text{Area} \times \text{Time} \times \text{Metal density})}$$

Where mass loss is expressed in mg, the area is expressed in cm² of the metal surface exposed, time is expressed in the hours of exposure & metal density is expressed in gm/cm³²⁰.

The inhibition efficiency was also determined using a thermometric technique²¹. These specimens 5.0*2.5*0.02 cm³ stagnant in a glass hook and immersed in a beaker containing 50 ml of the test solution at 23°C

with a precision of $\pm 0.5^\circ\text{C}$ were left exposed to air. Evaporation losses were made up with deionized water. After the test the specimens were cleaned with benzene. The duplicate experiments were performed in each case and the mean values of the mass loss were calculated.

Test solution preparation

The acidic solution was prepared by using de-ionized water. All the chemicals used were of analytical reagent quality. The temperature changes were measured at an interval of one minute using a thermometer with a precision of $\pm 0.5^\circ\text{C}$. The temperatures increased slowly at first then rapidly and attained a maximum temperature. The recorded percentage inhibition efficiencies were calculated as:

$$\eta = \frac{(RN_f - RN_i)}{RN_f} \times 100$$

Where RN_i and RN_f are the reaction number in the presence and absence of inhibitors respectively and RN ($^\circ\text{C}/\text{min}$) is defined as²²:

$$RN = \frac{(T_m - T_o)}{t}$$

Where T_m and T_o are the maximum and initial temperature, respectively and t is the time required to reach the maximum temperature.

Polarization techniques

The experimental set up used for the polarization measurements include a three-electrode cell and potentiostat (Elico Model CL-95) with a sweep generator was used. The saturated calomel electrode was used as a reference electrode. The variation of the mild steel electrode potential under open circuit conditions in the test solution were determined. Spontaneous reaction takes place on the metal surface due to a potential difference between cathodes and anodes when metal as an electrode is immersed in the aqueous environment, due to activation polarization both reaction rates decrease. The potential and corresponding current densities are called as corrosion potential (E_{corr}) and corrosion density (I_{corr}) respectively. Cathodic and anodic polarization of the working electrode and the curves shifts towards lower current densities with increasing the concentration of additives²³.

$$I_{corr} = \frac{\{\beta a \beta c\}}{2.303(\beta a + \beta c)} \times \frac{1}{Rp} = \left\{ \frac{\Delta i}{\Delta E} \right\}_{E_{corr}}$$

The values of βa and βc and Rp , I_{corr} can be calculated using the above equation.

The percentage inhibition efficiency ($\eta\%$) can be calculated as follows:

$$\eta\% = \frac{I - I_0}{I_0} \times 100$$

Where I and I_0 are the corrosion currents in uninhibited and inhibited solution respectively.

The corrosion rate can be calculated from the I_{corr} values using the relation:

$$CR (mmpy) = \frac{0.1288 \times I_{corr} \times E}{D}$$

$$\text{Current density} = \frac{\text{Current}}{\text{Area}}$$

Where I_{corr} is the corrosion current in $\mu\text{A}/\text{cm}^2$, E is the equivalent mass of the metal in mg and D is the density in gm/cm^3 ²⁴.

The corrosion rate was converted from mpy to mmpy using the following relation

$$1 \text{ mpy} = 0.0254 \text{ mmpy}$$

RESULTS AND DISCUSSION

The inhibition efficiency (%) calculated from the mass loss measurement for nitric acid media and inhibitors are given in Tables (1–4). It is observed that the inhibition efficiency increases with an increase in the concentration of the inhibitor (Fig. 1–4). The corrosion rate decreases with increases in the concentration of the inhibitors. Table 1 shows the maximum 93.43% inhibition efficiency for mild steel in 0.1N nitric acid solution whereas 0.5N shows 94.79%, respectively (Table 2). The Fig.1 and 2 confirm above-mentioned results.

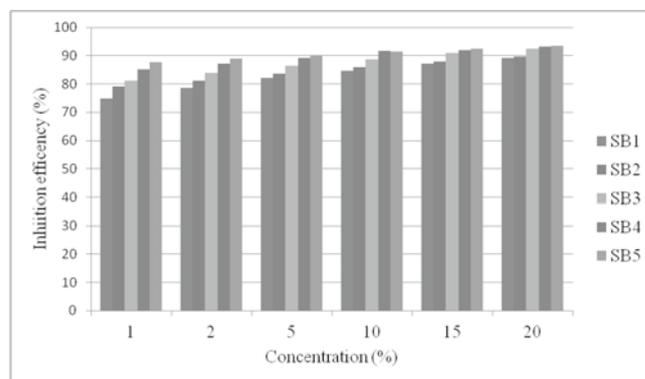


Figure 1. Inhibition efficiency v/s concentration of mild steel in 0.1N HNO₃

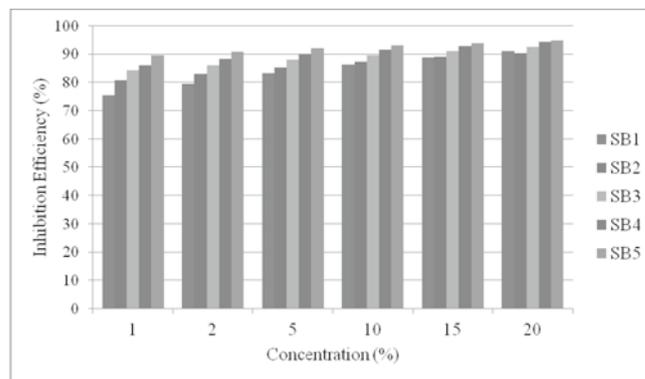


Figure 2. Inhibition efficiency v/s concentration of mild steel in 0.5N HNO₃

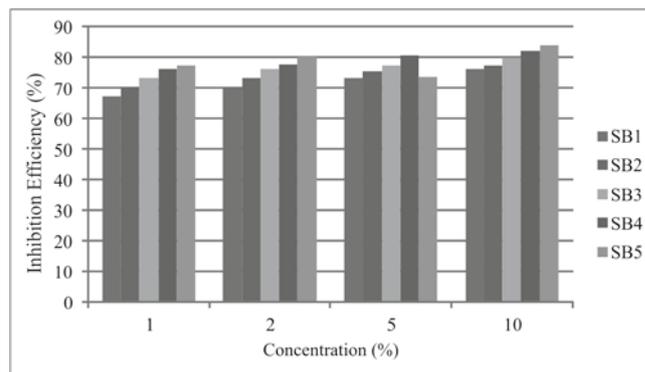


Figure 3. Inhibition efficiency v/s concentration of mild steel in 0.1N HNO₃ (Polarization)

Table 1. Effect on Schiff bases on mass loss data for corrosion of mild steel in 0.1 N nitric acid. Effective area of specimen: 3.875 cm². Temperature: 23±0.5°C. Immersion Time: 72 hours

Inhibitors Concentration [mM]	Mass loss [mg]	Inhibition Efficiency [η %]	Corrosion rate [mmpy]	Surface Coverage [θ]	Log [$\theta / 1-\theta$]
Blank	1570.25		381.88		
SB1					
1	394.68	74.87	95.99	0.7487	0.4740
2	333.61	78.75	81.13	0.7875	0.5690
5	279.74	82.18	68.03	0.8218	0.6640
10	238.94	84.78	58.11	0.8478	0.7460
15	200.71	87.22	48.81	0.8722	0.8340
20	169.92	89.18	41.32	0.8918	0.9160
SB2					
1	329.40	79.02	80.11	0.7902	0.5760
2	294.29	81.26	71.58	0.8126	0.6370
5	255.23	83.75	62.07	0.8375	0.7120
10	220.87	85.93	53.72	0.8593	0.7860
15	190.46	87.87	46.32	0.8787	0.8600
20	163.03	89.62	39.66	0.8962	0.9360
SB3					
1	294.32	81.26	71.58	0.8126	0.6370
2	252.29	83.93	61.36	0.8393	0.7180
5	211.85	86.51	51.52	0.8651	0.8070
10	175.58	88.82	42.70	0.8882	0.9000
15	143.65	90.85	34.94	0.9085	0.9970
20	119.30	92.40	29.01	0.9240	1.0850
SB4					
1	232.93	85.17	56.65	0.8517	0.7590
2	200.71	87.22	48.81	0.8722	0.8340
5	169.92	89.18	41.32	0.8918	0.9160
10	145.77	91.72	35.45	0.9172	0.9900
15	124.48	92.07	30.27	0.9207	1.0650
20	107.68	93.14	26.19	0.9314	1.1300
SB5					
1	192.79	87.72	46.89	0.8772	0.8540
2	172.37	89.02	41.92	0.8902	0.9090
5	151.97	90.32	36.99	0.9032	0.9700
10	133.75	91.48	32.53	0.9148	1.0310
15	117.54	92.51	28.59	0.9251	1.0920
20	103.15	93.43	25.09	0.9343	1.1530

The following order of the inhibition efficiency has been observed for five Schiff bases for mild steel in nitric acid for mass loss and the potentiostatic method. SB1 < SB2 < SB3 < SB4 < SB5

However, the values of inhibition efficiency obtained from the corrosion current (I_{corr}) values are lower than those obtained from the mass loss data. Similar observations have been reported earlier Tables (3–4). The potentiostatic polarization results for mild steel in 0.1 N

nitric acid solution shows 83.97% inhibition efficiency while 0.5 N acid solution shows 87.32%. The same results are presented in Figs. 3 and 4.

The order of the corrosion rate for mild steel in different test solutions observed by mass loss and potentiostatic polarization method is in good agreement but the corrosion rate calculated from the potentiostatic method is higher than that of the mass loss method. It is expected that the corrosion rate is maximum in the beginning and decreases when equilibrium is attained. However, contrary to the earlier studies^{25,26}, the corrosion rates obtained from potentiostatic measurements are not significantly higher than those obtained by the mass loss methods.

The variation of the reaction number with the inhibitor concentration indicates that the reaction number decreases with increasing the inhibitor concentration (Table 5). The thermometric methods show the 96.78% inhibition efficiency for 1 N nitric acid solution while the 2 N nitric acid solution shows 97.98% whereas 4 N nitric acid solution shows 98.32% inhibition efficiency, respectively.

This process blocks active sites, hence decreases the corrosion rate. In the present study, it is assumed that

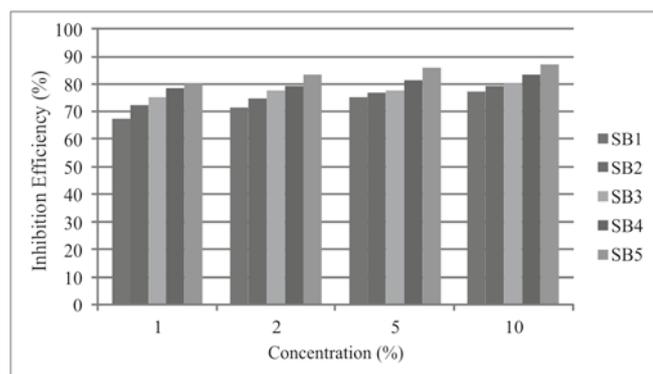
**Figure 4.** Inhibition efficiency v/s concentration of mild steel in 0.1 N HNO₃ (Polarization)

Table 2. Effect on Schiff bases on mass loss data for corrosion of mild steel in 0.5N nitric acid. Effective area of specimen: 3.875 cm². Temperature: 23±0.5°C. Immersion Time: 72 hours

Inhibitors Concentration [mM]	Mass loss [mg]	Inhibition Efficiency [η %]	Corrosion rate [mmpy]	Surface Coverage [θ]	Log [$\theta / 1-\theta$]
Blank	3455.35		840.34		
SB1					
1	844.77	75.55	205.45	0.7555	0.4900
2	705.26	79.59	171.52	0.7959	0.5910
5	574.75	83.37	139.78	0.8337	0.7000
10	472.72	86.32	114.96	0.8632	0.8000
15	386.36	88.82	93.96	0.8882	0.9000
20	304.40	91.19	74.03	0.9119	1.0150
SB2					
1	662.33	80.83	161.08	0.8083	0.6250
2	283.64	83.11	141.94	0.8311	0.6920
5	511.57	85.19	124.41	0.8519	0.7600
10	436.37	87.37	106.13	0.8737	0.8400
15	375.44	89.13	91.31	0.8913	0.9140
20	335.81	90.28	81.67	0.9028	0.9680
SB3					
1	539.26	84.39	131.15	0.8439	0.7330
2	479.33	86.13	116.57	0.8613	0.7930
5	413.22	88.04	100.49	0.8804	0.8670
10	359.56	89.59	87.44	0.8959	0.9350
15	308.25	91.08	74.97	0.9108	1.0090
20	256.45	92.58	62.37	0.9258	1.0960
SB4					
1	479.33	86.13	116.57	0.8613	0.7930
2	401.63	88.38	97.88	0.8838	0.8810
5	340.02	90.16	82.69	0.9016	0.9620
10	291.24	91.57	70.83	0.9157	1.0360
15	248.90	92.80	60.53	0.9280	1.1100
20	206.41	94.30	50.20	0.9403	1.1970
SB5					
1	364.79	89.44	88.525	0.8944	0.9280
2	317.43	90.81	77.20	0.9081	0.9950
5	276.84	91.99	67.33	0.9199	1.0600
10	238.47	93.10	58.00	0.9310	1.1300
15	209.57	93.94	50.97	0.9394	1.1900
20	179.99	94.79	43.77	0.9479	1.2600

the Schiff bases are adsorbed on the metal surface and decrease the surface area available for cathodic and anodic reaction to take place. Adsorption shows the collection of adsorb on the surface due to a cohesive force of attraction.

According to the Fontana²⁷ for the more commonly used low carbon steel and steel alloys, <1 mpy is outstanding corrosion rate, 1–5 mpy is excellent, 5–20 mpy is good and can be used with caution, 20–50 is fair and can be used for good exposure, 50–200 mpy is poor and above 200 mpy is completely unacceptable.

But the corrosion rates are due to a significant contribution of nitrate salt available in water, hence the best promising results are obtained by decreasing the corrosion rate due to anisalidine derivatives.

CONCLUSIONS

1. Anisidine derivatives show a synergistic effect in controlling the corrosion of mild steel in acidic solution containing higher concentration of the inhibitor.

2. The protection film on the metal surface consists of mild steel-anisidine derivatives complex and mild steel oxide.

3. Anisidine derivatives form a complex on the anodic site of the metal surface through the oxygen atom and nitrogen atom of –OH, –CHO, >C = O, and –NH₂ group.

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Table 3. Potentiostatic polarization data for mild steel in 0.1 N nitric acid. Effective area of specimen: 3.875 cm². Temperature: 23±0.5°C

Inhibitor Concentration [mM]	OCP [-mV]	I _{corr} mA/cm ²	Inhibition Efficiency [η%]	Corrosion Rate [mmpy]	β _a mV/decade	β _c -mV/decade	R _p [ohms]
Blank	583	912.65		416.73	80	147	0.0246
SB1							
1	575	298.25	67.32	136.19	85	149	0.0788
2	574	271.51	70.25	123.98	86	149	0.0872
5	573	244.43	73.25	111.47	87	150	0.0979
10	572	218.12	76.10	99.60	89	152	0.1117
SB2							
1	577	270.78	70.33	123.64	83	151	0.0859
2	570	244.13	73.25	111.47	87	153	0.0986
5	569	222.14	75.66	101.43	85	154	0.1071
10	571	206.99	77.32	94.51	90	154	0.1192
SB3							
1	569	243.40	73.33	101.14	87	152	0.0987
2	569	217.03	76.22	99.10	87	155	0.1115
5	568	206.99	77.32	94.51	90	153	0.1189
10	560	182.44	80.01	83.30	92	154	0.1371
SB4							
1	565	216.02	76.33	98.64	88	152	0.1120
2	562	202.15	77.85	92.30	89	154	0.1212
5	560	176.87	80.62	80.76	91	157	0.1414
10	562	162.91	82.15	74.39	94	158	0.1571
SB5							
1	562	206.72	77.35	94.39	90	150	0.1182
2	564	180.52	80.43	82.43	90	153	0.1363
5	560	161.08	73.55	73.55	93	155	0.1567
10	561	140.09	83.97	72.97	95	159	0.1843

Table 4. Potentiostatic polarization data for mild steel in 0.5 N nitric acid. Effective area of specimen: 3.875 cm². Temperature: 23±0.5°C.

Inhibitor Concentration [mM]	OCP [-mV]	I _{corr} mA/cm ²	Inhibition Efficiency [η%]	Corrosion Rate [mmpy]	β _a mV/decade	β _c -mV/decade	R _p [ohms]
Blank	584	1977.65		903.01	82	148	0.0116
SB1							
1	573	647.68	67.25	295.74	87	152	0.0371
2	570	566.60	71.35	258.71	88	153	0.0428
5	573	488.08	75.32	222.86	90	155	0.0507
10	572	449.92	77.25	205.44	89	156	0.0547
SB2							
1	569	546.82	72.35	249.68	86	152	0.0436
2	568	494.61	74.99	225.84	89	154	0.0495
5	567	457.23	76.88	208.78	91	157	0.0547
10	565	408.98	79.32	186.74	92	159	0.0627
SB3							
1	560	489.67	75.24	223.59	89	150	0.0495
2	555	442.00	77.65	201.82	90	156	0.0561
5	554	442.00	77.65	201.82	92	157	0.0570
10	550	389.20	80.32	177.71	94	159	0.0659
SB4							
1	559	422.23	78.65	192.79	91	151	0.0584
2	555	408.78	79.33	186.65	93	158	0.0622
5	550	370.81	81.25	169.32	94	159	0.0692
10	547	329.67	83.33	150.53	95	157	0.0780
SB5							
1	546	389.00	80.33	177.62	92	155	0.0644
2	550	329.87	83.32	150.62	94	159	0.0778
5	543	277.07	85.99	126.51	94	155	0.0917
10	544	250.77	87.32	114.50	96	157	0.1032

Table 5. Thermometric data for mild steel in 0.1 N, 2.0 N and 4.0 N nitric acid in presence of Schiff bases. Initial Temperature: $23 \pm 0.5^\circ\text{C}$. Area of Specimen: 3.875 cm^2

Inhibitor Concentration [mM]	1 N		2 N		4 N	
	RN	η , %	RN	η , %	RN	η , %
Blank	0.6830		0.9450		0.1710	
SB1						
1	0.1427	79.10	0.1758	81.40	0.0270	84.20
2	0.1205	82.35	0.1361	85.60	0.0230	86.53
5	0.1003	85.32	0.1143	87.90	0.0168	90.20
10	0.0818	88.02	0.1009	89.32	0.0120	93.00
15	0.0669	90.21	0.0830	91.22	0.0101	94.10
20	0.0522	92.35	0.0723	92.35	0.0074	95.70
SB2						
1	0.1228	88.02	0.1503	84.10	0.0233	86.35
2	0.1023	85.02	0.1162	87.70	0.0148	91.32
5	0.0880	87.12	0.0936	90.10	0.0137	92.01
10	0.0750	89.02	0.0699	92.60	0.0103	93.98
15	0.0591	91.35	0.0491	94.80	0.0068	96.01
20	0.0461	93.25	0.0463	95.10	0.0057	96.65
SB3						
1	0.1048	84.65	0.1238	86.90	0.0199	88.35
2	0.0871	87.25	0.0973	89.70	0.0148	91.35
5	0.0727	89.35	0.0756	92.00	0.0119	93.03
10	0.0591	91.35	0.0586	93.80	0.0097	94.32
15	0.0522	92.35	0.0435	95.40	0.0082	95.21
20	0.0402	94.12	0.0369	96.10	0.0057	96.65
SB4						
1	0.0871	87.25	0.1030	89.10	0.0148	91.32
2	0.0736	89.23	0.0728	92.30	0.0115	93.25
5	0.0591	91.35	0.0558	94.10	0.0072	95.80
10	0.0464	93.21	0.0506	94.65	0.0051	97.00
15	0.0365	94.65	0.0389	95.88	0.0036	97.88
20	0.0318	95.35	0.0312	96.70	0.0031	98.21
SB5						
1	0.0682	90.02	0.0680	92.80	0.0131	92.32
2	0.0591	91.34	0.0567	94.00	0.0097	94.32
5	0.0463	93.22	0.0397	95.80	0.0062	96.35
10	0.0395	94.22	0.0350	96.30	0.0055	96.78
15	0.0318	95.35	0.0273	97.11	0.0032	98.10
20	0.0220	96.78	0.0191	97.98	0.0029	98.32

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