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SYNTHESIS AND BIOLOGICAL ACTIVITY OF SOME NEW 4-THIAZOLIDINONES

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ABSTRACT : New heterocyclic compounds are of major importance because of growing problem of bacteria and fungi resistance and antimicrobial thiazolidinone have been gaining a lot of interest. A series of Thiazolidine-4-ones derivatives as antimicrobial agent were synthesized and screened for antibacterial activity against *S. aureus, E. coli* and *P. vulgaris* and antifungal activity against *A. fumigatus*, *C. albicans* and *C. albicans* ATCC. The structure of the newly synthesised compounds have been confirmed from elemental analysis, IR and ^{1}HNMR data.

Key words : Antibacterial activity, antifungal activity, thiazolidinones.

INTRODUCTION

With respect to a variety of biological activities heterocyclic compounds occupy nearly the first place among the other classes of organic compounds. The thiazolidinone have emerged as antimicrobial agents of immense interest because of their broad spectrum of in vitro activity and their in vivo chemotherapeutic activity (Patel and Parekh, 1988; Trivedi *et al*, 2004; Omar *et al,* 2010). Based on these findings, and in continuation of our drug research program concerning synthesis of new safer and more biologically active thiazolidinones (Saleh *et al,* 2013; Nawale and Dhake, 2012; Patel and Shaikh, 2010; Shrivastava *et al,* 2012). It was of synthesize a new series of thiazolidinone derivatives with the hope to obtain more active and less toxic antimicrobial agents. Thiazolidinones have been reported to be biologically versatile compound posses variety of activity including anti-inflammatory activity (Mishra *et al,* 1997), antitubercular activity (Thaker *et al,* 2003), antifungal (Patel and Mehta, 2006) and antibacterial activity (Hogale and Uthale, 1990). Being involved in a research program aiming at finding out new structure leads that would act as potent antimicrobial agents, we have reported the synthesis and antimicrobial activity of some lead compounds comprising mainly the thiazolidinone moiety substituted with various functionalities and attached to defferent heterocyclic ring system through various linkage.

MATERIALS AND METHODS

Chemistry

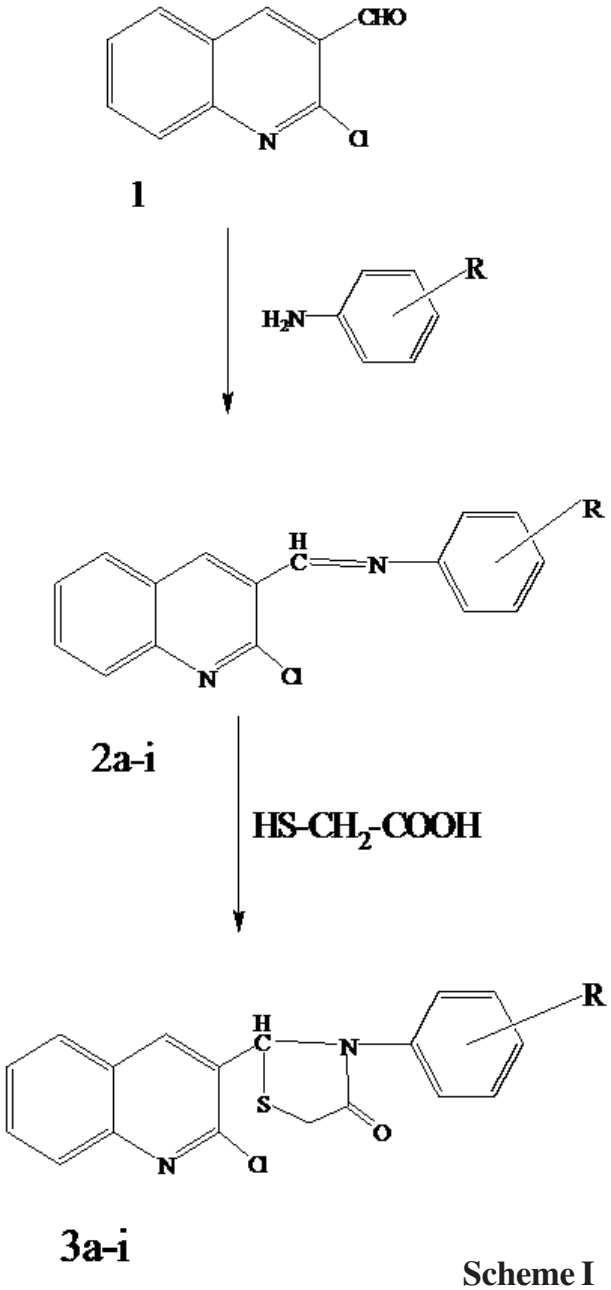
The melting points of compounds were determined in open capillaries and are uncorrected. The homogeneity of the synthesized compounds were routinely checked by thin layer chromatography on silica gel G plates. The IR spectra were recorded on a Beckman Acculab-10 Spectrometer (í max in cm^{-1}) and the ^{1}HNMR spectra were recorded by Brucker DPX-300MHz using $CDCl_3$ as solvent.

Biological evaluation (Antimicrobial activity)

All the synthesized compounds were tested for their antimicrobial activity. The antimicrobial activity was assayed by using cup plate method (Chuinkshank *et al,* 1975). The antibacterial activities of compounds were tested against Staphlococcus aureus, Escherischia coli, Proteus vulgaris. The antibacterial activity of the synthesized compounds was tested against strains isolated from animal by products and were accused of being a direct cause of food in toxication in human. The antifungal activities of the compounds were tested against *Aspergillus fumigatus, Candida albicans* and *Candida albicans* ATCC. The screening results were compared with Ampicillin and gattifloxacin for antibacterial and fluconazole for antifungal activities respectively. The results were recorded for each tested compounds as the average diameter of Inhibition Zone (IZ) of microbial growth around the disc in mm.

RESULTS AND DISCUSSION

All the compounds reported in table 1 were tested in vitro for their antimicrobial activity against various microbes. Compound 1 exhibited mild antibacterial activity. Among the compounds 2a-2i showed moderate



Scheme I

R = H, 2-OH, 4-OCH_3, 4-OH & 3-OCH_3, 2-Cl, 4-Cl, 2, 4-Cl, 2, 6-Cl, 4-$N(CH_3)_2$

antimicrobial activity. Cyclization of compounds 2a-2i with thioglycolic acid yielded compounds 3a-3i. Among the compounds 3a-3i, compound 3g and 3h exhibited good antibacterial activity against S. aureus, E.coli and P. vulgaris. Moreover compound 3h and 3i showed good antifungal activity against different fungi. Compounds 3d, 3e, 3f and 3i have shown significant antimicrobial activity.

Preparation of 2-chloroquinoline-3-carboxaldehyde I.

Dimethylformamide (1.0 mol) was cooled to 0^0 C in a flask equipped with a tube and phosphoryl chloride (1.5 mol) was added acetanilide (0.1 mol) and the solution was heated under reflux for 16 hr. at room temp. The reaction mixture was isolated and recrystallised from ethyl acetate. Yield 68%, m.p. 148^0C. IR (KBr) í max in cm^{-1} : 3150 (Aromatic CH str.), 1624 (C=C of aromatic ring), 1607 (C=N), 1506 (C-N), 760 (C-Cl). ^{1}HNMR ($CDCl_3$+DMSO-d_6) ä in ppm: 8.70 (s, 1H, N=CH), 7.70-8.30 (m, 10H, Ar-H), Anal. Calcd. for $C_{10}H_6NOCl$ C, 62.68; H, 3.16; N, 7.31; Found: C, 62.65; H, 3.14; N, 7.30%.

Table 1 : Antibacterial and antifungal activity of the compounds **2a-2i** and compounds **3a-3i.**

Comp.No.	R	Bacterial growth inhibition (diameter)			Fungal growth inhibition(diameter)		
		S. aureus	*E. coli*	*P. vulgaris*	*A. fumigatus*	*C. albicans*	*C. albicans* ATCC
2a	H	5mm	-	6mm	-	-	-
2b	2-OH	-	-	-	8 mm	-	6mm
2c	4-OCH_3	8mm	9mm	8mm	-	6mm	-
2d	4-OH,3-OCH_3	-	-	9mm	9mm	8mm	-
2e	2-Cl	10mm	10mm	-	-	-	8mm
2f	4-Cl	9mm	-	-	-	-	-
2g	2,4-Cl	11mm	-	11mm	11mm	10mm	12mm
2h	2,6-Cl	-	10mm	10mm	10mm	9mm	10mm
2i	$N(CH_3)_2$	12mm	-	-	-	-	-
3a	H	-	11mm	12mm	-	12mm	18mm
3b	2-OH	12mm	10mm	-	11mm	-	17mm
3c	4-OCH_3	14mm	-	15mm	-	16mm	-
3d	4-OH,3-OCH_3	16mm	18mm	15mm	17mm	19mm	19mm
3e	2-Cl	-	15mm	-	21mm	-	22mm
3f	4-Cl	19mm	-	16mm	-	22mm	23mm
3g	2,4-Cl	25mm	20mm	22mm	23mm	18mm	-
3h	2,6-Cl	24mm	23mm	21mm	20mm	25mm	23mm
3i	$N(CH_3)_2$	22mm	21mm	-	25mm	30mm	26mm
Ampicillin		20mm	18mm	18mm			
gattifloxacin		25mm	22mm	20mm			
fluconazol						29mm	25mm

Preparation of substituted aryl-2-chloroquinoline-3-yl azomethine (2a-2i).

2-chloroquinoline-3-carboxaldehyde (0.1 mol) was refluxed with different amines (0.1 mol) in suitable solvent on water bath for 6 hr. methanol as solvent. The reaction mixture was poured in to crushed ice. The product was isolated and recrystallised from ethanol to yield compounds (2a-2i).

N-((2-chloroquinoline-3-yl)methylene)aniline (2a).

Yield 65% ,m.p. 159^{0}C. IR (KBr) í max in cm^{-1} : 3150 (Aromatic CH str.), 1624 (C=C of aromatic ring), 1608 (C=N), 1509 (C-N), 763 (C-Cl). ^{1}HNMR ($CDCl_3$+DMSO-d_6) ä in ppm: 8.70 (s, 1H, N=CH), 7.72-8.30 (m, 10H, Ar-H). Anal. Calcd. for $C_{16}H_{11}N_2Cl$ C, 62.68; H, 3.16; N, 7.31; Found: C, 62.65; H, 3.14; N, 7.30%.

2-((2-chloroquinoline-3-yl)methyleneamino)phenol (2b).

Yield 61% ,m.p. 135^{0}C. IR (KBr) í max in cm^{-1} : 3428 (OH), 3150 (Aromatic CH str.), 1614 (C=C of aromatic ring), 1607 (C=N), 1506 (C-N), 760 (C-Cl). ^{1}HNMR ($CDCl_3$+DMSO-d_6) ä in ppm: 12.50 (s, OH exchangeable with D_2O), 8.70 (s, 1H, N=CH), 7.70-8.32 (m, 9H, Ar-H). Anal. Calcd. for $C_{16}H_{11}N_2ClO$ C, 67.97; H, 3.92; N, 9.91; Found: C, 67.95; H, 3.94; N, 9.90%

N-((2-chloroquinoline-3-yl)methylene)-4-methoxyaniline (2c).

Yield 60% , m.p. 160^{0}C. IR (KBr) í max in cm^{-1} : 3150 (Aromatic CH str.) , 1614 (C=C of aromatic ring), 1607 (C=N), 1506 (C-N), 1228 (OCH_3), 760 (C-Cl). ^{1}HNMR ($CDCl_3$+DMSO-d_6) ä in ppm: 8.78 (s, 1H, N=CH), 7.72-8.31 (m, 9H, Ar-H), 3.43 (s, 3H, OCH_3). Anal. Calcd. for $C_{17}H_{13}N_2ClO$ C, 68.81; H, 4.42; N, 9.44; Found: C, 68.65; H, 4.44; N, 9.40%.

N-((2-chloroquinoline-3-yl)methylene)-2-methoxyphenol (2d).

Yield 59% , m.p. 166^{0}C. IR (KBr) í max in cm^{-1} : 3150 (Aromatic CH str.), 1615 (C=C of aromatic ring), 1609 (C=N), 1504 (C-N), 1227 (OCH_3), 760 (C-Cl). ^{1}HNMR ($CDCl_3$+DMSO-d_6) ä in ppm: 12.50 (s, OH exchangeable with D_2O), 8.78 (s, 1H, N=CH), 7.70-8.31 (m, 8H, Ar-H), 3.46 (s, 3H, OCH_3). Anal. Calcd. for $C_{17}H_{13}N_2ClO_2$ C, 65.29; H, 4.19; N, 8.96; Found: C, 68.65; H, 4.44; N, 9.40%.

2-chloro-N-((2-chloroquinolin-3-yl)methylene)aniline (2e).

Yield 58% ,m.p. 158^{0}C. IR (KBr) í max in cm^{-1} : 3150 (Aromatic CH str.), 1619 (C=C of aromatic ring), 1607 (C=N), 1504 (C-N), 760 (C-Cl). ^{1}HNMR ($CDCl_3$+DMSO-d_6) ä in ppm: 8.70 (s, 1H, N=CH), 7.73-8.30 (m, 9H, Ar-H). Anal. Calcd. for $C_{16}H_{10}N_2Cl_2$ C, 63.81; H, 3.35; N, 9.30; Found: C, 63.85; H, 3.34; N, 9.34%.

4-chloro-N-((2-chloroquinolin-3-yl)methylene)aniline (2f).

Yield 56% ,m.p. 159^{0}C. IR (KBr) í max in cm^{-1} : 3150 (Aromatic CH str.), 1630 (C=C of aromatic ring), 1603 (C=N), 1506 (C-N), 760 (C-Cl). ^{1}HNMR ($CDCl_3$+DMSO-d_6) ä in ppm: 8.70 (s, 1H, N=CH), 7.70-8.32 (m, 9H, Ar-H). Anal. Calcd. for $C_{16}H_{10}N_2Cl_2$ C, 63.81; H, 3.35; N, 9.30; Found: C, 63.85; H, 3.34; N, 9.34%.

2,4-dichloro-N-((2-chloroquinolin-3-yl)methylene)aniline (2g).

Yield 54% ,m.p. 189^{0}C. IR (KBr) í max in cm^{-1} : 3150 (Aromatic CH str.), 1614 (C=C of aromatic ring), 1607 (C=N), 1505 (C-N), 760 (C-Cl). ^{1}HNMR ($CDCl_3$+DMSO-d_6) ä in ppm: 8.70 (s, 1H, N=CH), 7.72-8.30 (m, 8H, Ar-H). Anal. Calcd. for $C_{16}H_9N_2Cl_3$ C, 57.26; H, 2.70; N, 8.35; Found: C, 57.25; H, 2.74; N, 8.34%.

2,6-dichloro-N-((2-chloroquinolin-3-yl)methylene)aniline (2h).

Yield 53% ,m.p. 190^{0}C. IR (KBr) í max in cm^{-1} : 3150 (Aromatic CH str.) 1614 (C=C of aromatic ring), 1603 (C=N), 1506 (C-N), 760 (C-Cl). ^{1}HNMR ($CDCl_3$+DMSO-d_6) ä in ppm: 8.70 (s, 1H, N=CH), 7.70-8.31 (m, 8H, Ar-H). Anal. Calcd. for $C_{16}H_9N_2Cl_3$ C, 57.26; H, 2.70; N, 8.35; Found: C, 57.25; H, 2.74; N, 8.34%.

N^1-((2-chloroquinoline-3-yl)methylene)-N^4,N^4-dimethylbenzene-1,4-diamine (2i).

Yield 50% ,m.p. 198^{0}C. IR (KBr) í max in cm^{-1} : 3150 (Aromatic CH str.), 1618 (C=C of aromatic ring), 1607 (C=N), 1503 (C-N), 760 (C-Cl). ^{1}HNMR ($CDCl_3$+DMSO-d_6) ä in ppm: 8.70 (s, 1H, N=CH), 7.72-8.30 (m, 10H, Ar-H), 1.35 (s,6H, $N(CH_3)_2$). Anal. Calcd. for $C_{18}H_{16}N_3Cl$ C, 69.79; H, 5.21; N, 13.56; Found: C, 69.75; H, 5.19; N, 13.60%.

Preparation of 2-(2-chloroquinolin-3-yl)-3-(substitutedphenyl)thiazolidin-4-one(3a-3i).

To ethanolic solution (60 ml) of compounds (2a-2i) (0.1mol) thioglycolic acid (0.1mol) was added in the presence of anhydrous zinc chloride. The reaction mixtures were refluxed for 10 h. The excess of solvent was distilled off and separated masses were poured in to ice water, filtered and washed with water and recrystallized from appropriate solvents to give compounds (3a-3i).

2-(2-chloroquinolin-3-yl)-3-phenylthiazolidin-4-one (3a)

Yield 49% ,m.p. 190^{0}C. IR (KBr) í max in cm^{-1} : 3150 (Aromatic CH str.), 1674 (C=O str.), 1614 (C=C of aromatic ring), 1607 (C=N), 1506 (C-N), 790 (C-S-C of oxothiazole), 760 (C-Cl). ^{1}HNMR ($CDCl_3$+DMSO-d_6) ä in ppm: 12.50 (s, OH exchangeable with D_2O), 8.70 (s, 1H, N-CH), 7.70-8.33 (m, 10H, Ar-H), 3.75 (s, 2H, CH_2 of oxothiazole). Anal. Calcd. for $C_{18}H_{13}N_2ClOS$ C, 63.43; H, 3.84; N, 8.22; Found: C, 63.45; H, 3.87; N, 8.22%

2-(2-chloroquinolin-3-yl)-3-(2-hydroxyphenyl)thiazolidin-4-one (3b)

Yield 48% ,m.p. 199^{0}C. IR (KBr) í max in cm^{-1} : 3150 (Aromatic CH str.), 1674 (C=O str.), 1614 (C=C of aromatic ring), 1605 (C=N), 1508 (C-N), 794 (C-S-C of oxothiazole), 764 (C-Cl). ^{1}HNMR ($CDCl_3$+DMSO-d_6) ä in ppm: 12.50 (s, OH exchangeable with D_2O), 8.70 (s, 1H, N-CH), 7.70-8.30 (m, 10H, Ar-H), 3.75 (s, 2H, CH_2 of oxothiazole). Anal. Calcd. for $C_{18}H_{13}N_2ClO_2S$ C, 60.59; H, 3.67; N, 7.85; Found: C, 60.55; H, 3.64; N, 7.83%

2-(2-chloroquinolin-3-yl)-3-(4-methoxyphenyl)thiazolidin-4-one (3c)

Yield 46% ,m.p. 201^{0}C. IR (KBr) í max in cm^{-1} : 3150 (Aromatic CH str.), 1674 (C=O str.), 1614 (C=C of aromatic ring), 1607 (C=N), 1506 (C-N), 1228 (OCH_3), 790 (C-S-C of oxothiazole), 760 (C-Cl). ^{1}HNMR ($CDCl_3$+DMSO-d_6) ä in ppm: 8.70 (s, 1H, N-CH), 7.70-8.30 (m, 9H, Ar-H), 3.75 (s, 2H, CH_2 of oxothiazole), 3.46 (s, 3H, OCH_3). Anal. Calcd. for $C_{19}H_{15}N_2ClO_2S$ C, 61.53; H, 4.08; N, 7.55; Found: C, 61.55; H, 4.04; N, 7.53%

2-(2-chloroquinolin-3-yl)-3-(4-hydroxy-3-methoxyphenyl)thiazolidin-4-one (3d)

Yield 45% ,m.p. 208^{0}C. IR (KBr) í max in cm^{-1} : 3150 (Aromatic CH str.) 1674 (C=O str.), 1614 (C=C of aromatic ring), 1609 (C=N), 1503 (C-N), 1228 (OCH_3), 794 (C-S-C of oxothiazole), 760 (C-Cl). ^{1}HNMR ($CDCl_3$+DMSO-d_6) ä in ppm: 12.50 (s, OH exchangeable with D_2O), 8.70 (s, 1H, N-CH), 7.70-8.30 (m, 8H, Ar-H), 3.75 (s, 2H, CH_2 of oxothiazole), 3.46 (s, 3H, OCH_3). Anal. Calcd. for $C_{19}H_{15}N_2ClO_3S$ C, 58.99; H, 3.91; N, 7.24;

Found: C, 58.95; H, 3.94; N, 7.23%

3(2-chlorophenyl)-2-(2-chloroquinolin-3-yl)thiazolidin-4-one (3e)

Yield 43% ,m.p. 212^{0}C. IR (KBr) í max in cm^{-1} : 3150 (Aromatic CH str.) 1674 (C=O str.), 1506 (C-N),1607 (C=N), 1614 (C=C of aromatic ring), 790 (C-S-C of oxothiazole),760 (C-Cl). ^{1}HNMR ($CDCl_3$+DMSO-d_6) ä in ppm: 12.50 (s, OH exchangeable with D_2O), 8.70 (s, 1H, N-CH), 7.70-8.30 (m, 9H, Ar-H), 3.75 (s, 2H, CH_2 of oxothiazole). Anal. Calcd. for $C_{18}H_{12}N_2Cl_2OS$ C, 57.61; H, 3.22; N, 7.46; Found: C, 57.65; H, 3.27; N, 7.42%

3(4-chlorophenyl)-2-(2-chloroquinolin-3-yl)thiazolidin-4-one (3f)

Yield 42% ,m.p. 213^{0}C. IR (KBr) í max in cm^{-1} : 3150 (Aromatic CH str.), 1674 (C=O str.), 1614 (C=C of aromatic ring), 1607 (C=N), 1506 (C-N), 790 (C-S-C of oxothiazole), 760 (C-Cl). ^{1}HNMR ($CDCl_3$+DMSO-d_6) ä in ppm: 12.53 (s, OH exchangeable with D_2O), 8.70 (s, 1H, N-CH), 7.72-8.30 (m, 9H, Ar-H), 3.75 (s, 2H, CH_2 of oxothiazole). Anal. Calcd. for $C_{18}H_{12}N_2Cl_2OS$ C, 57.61; H, 3.22; N, 7.46; Found: C, 57.65; H, 3.27; N, 7.42%

2-(2-chloroquinolin-3-yl)-3-(2,4-dichlorophenyl)thiazolidin-4-one (3g)

Yield 41% ,m.p. 220^{0}C. IR (KBr) í max in cm^{-1} : 3150 (Aromatic CH str.) 1674 (C=O str.), 1614 (C=C of aromatic ring), 1607 (C=N), 1506 (C-N), 793 (C-S-C of oxothiazole), 762 (C-Cl). ^{1}HNMR ($CDCl_3$+DMSO-d_6) ä in ppm: 8.73 (s, 1H, N-CH), 7.70-8.32 (m, 8H, Ar-H), 3.74 (s, 2H, CH_2 of oxothiazole). Anal. Calcd. for $C_{18}H_{11}N_2Cl_3OS$ C, 52.77; H, 2.71; N, 6.84; Found: C, 52.75; H, 2.73; N, 6.82%

2-(2-chloroquinolin-3-yl)-3-(2,6-dichlorophenyl)thiazolidin-4-one (3h)

Yield 39% ,m.p. 221^{0}C. IR (KBr) í max in cm^{-1} : 3150 (Aromatic CH str.) 1674 (C=O str.), 1614 (C=C of aromatic ring), 1607 (C=N), 1506 (C-N), 791 (C-S-C of oxothiazole),760 (C-Cl). ^{1}HNMR ($CDCl_3$+DMSO-d_6) ä in ppm: 8.70 (s, 1H, N-CH), 7.71-8.30 (m, 8H, Ar-H), 3.75 (s, 2H, CH_2 of oxothiazole). Anal. Calcd. for $C_{18}H_{11}N_2Cl_3OS$ C, 52.77; H, 2.71; N, 6.84; Found: C, 52.75; H, 2.73; N, 6.82%

2-(2-chloroquinolin-3-yl)-3-(4-(dimethyamino)phenyl)thiazolidin-4-one (3i)

Yield 37% ,m.p. 230^{0}C. IR (KBr) í max in cm^{-1} : 3150 (Aromatic CH str.) 1614 (C=C of aromatic ring), 1607 (C=N), 1506 (C-N), 763 (C-Cl). ^{1}HNMR ($CDCl_3$+DMSO-d_6) ä in ppm: 8.74 (s, 1H, N=CH), 7.70-8.33 (m, 9H, Ar-H),), 3.77 (s, 2H, CH_2 of oxothiazole), 1.35 (s,6H, $N(CH_3)_2$). Anal. Calcd. for $C_{20}H_{18}N_3Cl$ OS C, 62.57; H, 4.73; N, 10.95; Found: C, 62.55; H, 4.78; N, 10.94%.

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