Synthesis and Antimicrobial Activity of *N*-{4-[3-(4-Chlorophenyl)-oxiranecarbonyl]phenyl}-2-(1,1,3-trioxo-1,3-dihydrobenzo[*d*]isothiazol-2-yl)acetamide

A. I. EL-SHENAWY

Department of Chemistry, Faculty of Science, Benha University, Benha, Egypt
e-mail: ref_at@hotmail.com

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Epoxide derivative *II* was used to synthesize a number of new condensed and noncondensed heterocyclic systems. Thus, reaction of *II* with amines, hydrazines, active methylene compounds and Friedel—Crafts reactions have been studied. On the other hand, *II* reacted with Grignard reagents and afforded products with an open epoxide ring. However, *II* condensed with thiourea and glycine gave oxazolinethione and 2-morpholinone derivatives. Also, *II* reacted with acetic acid and gave hydroxypropanoyl derivative. Action of different nitriles with *II* gave the hydroxy amide derivatives, which underwent cyclization and afforded 2-oxazoline derivatives.

The reported pharmaceutical properties [1—4] of analgesic agents, enzyme inhibitors, and anxiolytic agent 1,1,3-trioxo-1,3-dihydrobenzo[*d*]isothiazole and its derivatives promoted our interest for the synthesis of the title compound and its derivatives and a variety of stabilized carbanions have been widely used for the intramolecular ring opening of 1,2-epoxides. Most commonly these carbanions are stabilized by adjacent electron-withdrawing groups (EWGs) such as cyano, sulfonyl or sulfur-containing groups [5]. When the EWG is a carbonyl, some ambiguity may result from the presence of two nucleophilic sites (C and O) which may intramolecularly displace the oxirane ring leading to the corresponding *C*- or *O*-alkylation products, respectively. Our program is to study the effect of bulky heteroaryl moiety at the β-position on the behaviour of the oxirane ring towards different nitrogen and carbon nucleophiles and its behaviour towards the expected biological activity of some synthesized compounds.

Treatment of an alcoholic solution of chalcone *I* with hydrogen peroxide in alkaline medium yielded *N*-{4-[3-(4-chlorophenyl)oxiranecarbonyl]phenyl}-2-(1,1,3-trioxo-1,3-dihydrobenzo[*d*]isothiazol-2-yl)acetamide (*II*) (Scheme 1).

It has been reported [6] that the oxirane ring of α,β-epoxy-β-aroylacrylic acid was opened by nucleophilic reagents. In the present investigation, the epoxide *II* was allowed to react with *p*-bromoaniline and benzylamine in boiling butan-1-ol and yielded *N*-{4-[3-(4-chlorophenyl)-3-arylamino-2-hydroxypropanoyl]-phenyl}-2-(1,1,3 -trioxo-1,3-dihydrobenzo[*d*]isothiazol-2-yl)acetamide *IIIa, IIIb*.

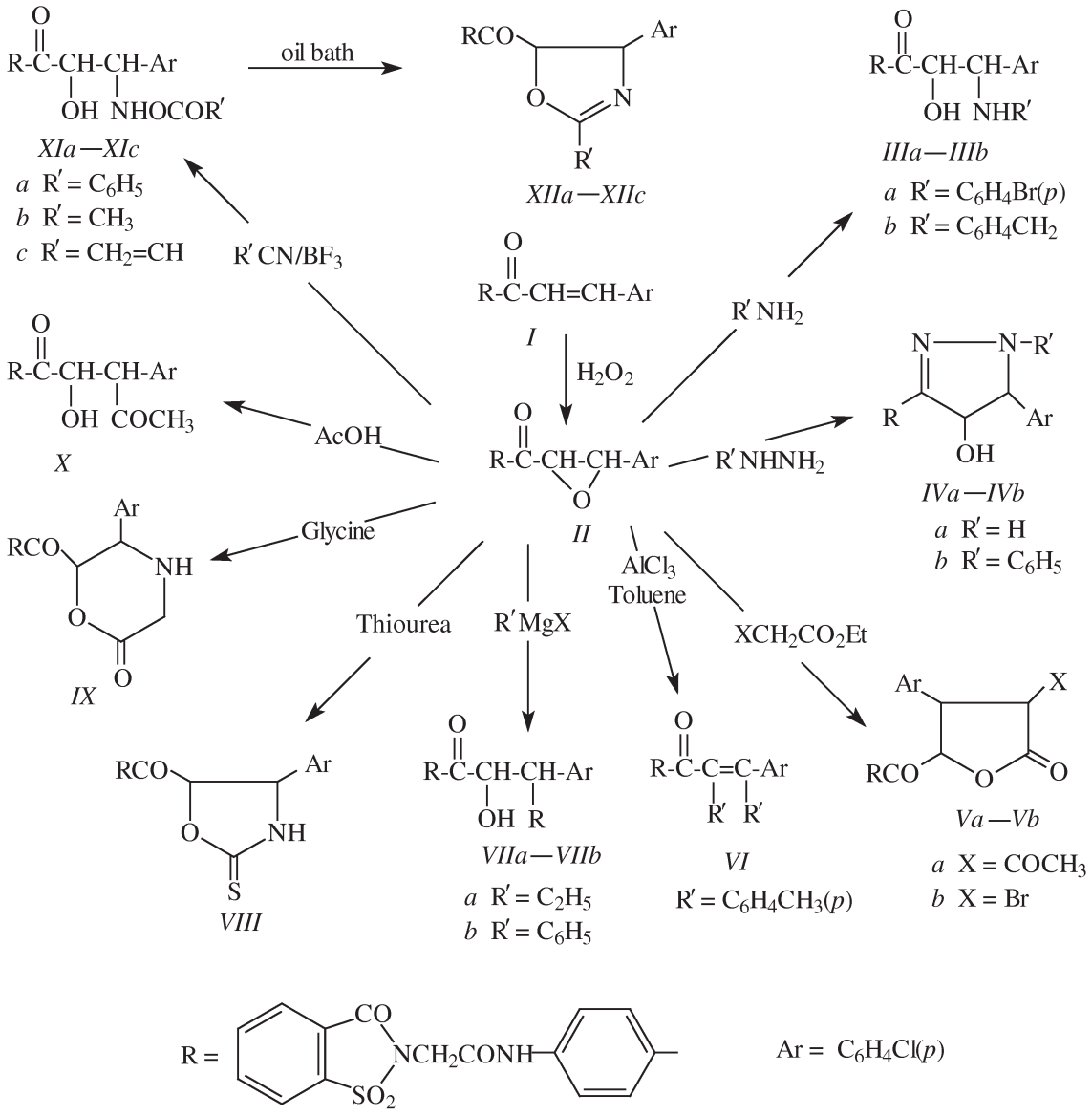
Recently, it has been shown [7] that the oxirane ring of α,β-epoxy ketone was opened by action of hydrazines. Thus, *II* was reacted with hydrazine hydrate and phenyl hydrazine and afforded *N*-{4-[5-(4-chlorophenyl)-4-hydroxy-4,5-dihydro-1*H*/1-phenyl-pyrazolin-3-yl]phenyl}-2-(1,1,3-trioxo-1,3-dihydrobenzo[*d*]isothiazol-2-yl)acetamide (*IVa, IVb*).

On the other hand, the reactivity of the oxirane ring towards active methylene compounds has been studied. The reaction of the epoxide *II* with ethyl acetoacetate or ethyl bromoacetate with the fission of the oxirane ring, followed by ring closure, furnished *N*-{4-[4-(4-chlorophenyl)-3-aceto/bromo-2-oxo-2,3,4,5-tetrahydrofuran-5-ylcarbonyl]phenyl}-2-(1,1,3-trioxo-1,3-dihydrobenzo[*d*]isothiazol-2-yl)acetamide (*Va, Vb*).

The reaction of epoxides with aromatic hydrocarbons in the presence of anhydrous aluminium chloride under Friedel—Crafts conditions has been reported [8]. Thus, toluene was arylated by using *II* and yielded *N*-{4-[3-(4-chlorophenyl)-2,3-di(*p*-tolyl)acryloyl]phenyl}-2-(1,1,3-trioxo-1,3-dihydrobenzo[*d*]isothiazol-2-yl)acetamide (*VI*).

Previously it was reported [9] that epoxides underwent ring opening by action of Grignard reagents. So, *II* was reacted with ethyl magnesium bromide and phenyl magnesium bromide and afforded *N*-{4-[3-(4-chlorophenyl)-3-ethyl/phenyl-2-hydroxypropanoyl]-phenyl}-2-(1,1,3-trioxo-1,3-dihydrobenzo[*d*]isothiazol-2-yl)acetamide (*VIIa, VIIb*).

Reactions of the epoxides with thiourea and glycine were reported [10—12]. So, condensation of epoxide *II* with thiourea and glycine in the presence of dimethylformamide (DMF) as a solvent and anhydrous aluminium chloride as a catalyst furnished *N*-{4-[4-(4-



Scheme 1

chlorophenyl)-2-thioxo-2,3,4,5-tetrahydrooxazolin-4-ylcarbonyl]phenyl}-2-(1,1,3-trioxo-1,3-dihydrobenzo[*d*]isothiazol-2-yl)acetamide (*VIII*) and *N*-{4-[5-(4-chlorophenyl)-2-oxo-2,3,5,6-tetrahydromorpholin-6-ylcarbonyl]phenyl}-2-(1,1,3-trioxo-1,3-dihydrobenzo[*d*]isothiazol-2-yl)acetamide (*IX*).

The reaction of the epoxide *II* with acetic acid at room temperature afforded *N*-{4-[3-(4-chlorophenyl)-3-acetyl-2-hydroxypropanoyl]phenyl}-2-(1,1,3-trioxo-1,3-dihydrobenzo[*d*]isothiazol-2-yl)acetamide (*X*).

The interest in the biological and industrial potential [13] of 2-oxazolines resulted in various synthetic procedures for the introduction of five-membered nitrogen and oxygen-containing heterocycles, *i.e.* 2-oxazoline into hydrocarbon chain. Different studies of the reactions of various short-chain epoxides with nitriles in the presence of a catalyst leading to the formation of 2-oxazolines have been described [14]. These observations prompted us to carry out the conversion of α,β-epoxy ketone *II* into 2-oxazolines *via* the reaction with benzonitrile, acetonitrile, and acrylonitrile in the presence of boron triflouride etherate as catalyst yielding the corresponding α-hydroxy-β-amido derivatives *XIa*—*XIc*, which underwent cyclization by subjection to fusion in an oil bath at 210—220 °C and furnished *N*-{4-[4-(4-chlorophenyl)-2-R′-4,5-dihydrooxazolin-5-ylcarbonyl]phenyl}-2-(1,1,3-trioxo-1,3-dihydrobenzo[*d*]isothiazol-2-yl)acetamides *XIIa*—*XIIc*.

The results of the antimicrobial activity are summarized in Table 1.

EXPERIMENTAL

Origin of cultures: Botany Department, Faculty of Science, Benha University, Egypt.

Table 1. Activity (A) and Minimum Inhibitory Concentration (MIC) ($\mu g/cm^3$)

Compound	*Aspergillus flavus*		*E.coli*		*Staphylococcus aureus*		*Bacillus circulans*	
	A	MIC	*A*	MIC	*A*	MIC	*A*	MIC
IIIa	+	250	–	–	+	250	++	125
IVb	++	125	+	250	+	250	+	250
VIb	++	250	–	–	–	–	++	250
VII	+	250	+	250	–	–	+	125
VIII	+	250	–	–	+	250	–	–
IX	++	125	+	250	++	125	+	250
XIa	+	250	–	–	+	125	+	125
XIb	++	125	++	250	+++	125	++	125
XIc	++	125	+	125	++	250	+	250
XIIc	++	250	++	250	++	250	++	125

The width of the zone of inhibition indicates the potency of antimicrobial activity, – no antimicrobial activity, + weak activity with diameter equal to 0.5—0.7 cm, ++ moderate activity with the diameter zone equal to 1.0—1.2 cm, +++ marked activity with the diameter zone equal to 1.6—1.8 cm.

All melting points are uncorrected. The IR spectra in KBr were recorded on a Shimadzu 470 spectrometer. The ^{1}H NMR spectra were measured on a Varian EM-390-90 MHz spectrometer using TMS as internal reference and the chemical shifts are expressed as δ. The mass spectra were recorded on HP Model: MS 5988 at 70 eV. The physical data are listed in Table 2.

N-{4-[3-(4-Chlorophenyl)ethyleneoxidecarbonyl]phenyl}-2-(1,1,3-trioxo-1,3-dihydrobenzo[*d*]isothiazol-2-yl)acetamide (*II*)

A solution of chalcone *I* (0.01 mol) in acetone (40 cm^3) was mixed with 8 % aqueous sodium hydroxide (12 cm^3) followed by addition of hydrogen peroxide (30 %, 5 cm^3). The solution was shaken and heated for 2 h, then allowed to stand overnight at room temperature, water was then added and the solution acidified with dilute HCl. The mixture was extracted with ether and the solid separated was crystallized from the proper solvent. The IR spectrum of *II* showed absorption bands at 1730—1750 cm^{-1} (ν(CO), cyclic imides), 1680 cm^{-1}(ν(CO), ketone), 1660 cm^{-1}(ν(CO), amide), 1320 and 1120 cm^{-1} ($\nu(SO_2)$), 1070 cm^{-1} (ν(C—O—C)), and 3150 cm^{-1} (ν(NH)). The mass spectrum showed peaks at m/z (relative intensity/%): 496.5 (0.20) (M^+), 372 (0.50), 371 (0.11), 374 (0.17), 315 (2.15), 196 (100.00), 183 (2.60), 168 (6.70), 141 (2.95), 140 (8.30), 99 (41.10), 91 (30.40), 77 (36.50), 75 (13.50), 65 (21.20), 57 (44.30).

N-{4-[3-(4-Chlorophenyl)-3-arylamino-2-hydroxypropanoyl]phenyl}-2-(1,1,3-trioxo-1,3-dihydrobenzo[*d*]isothiazol-2-yl)acetamide *IIIa, IIIb*

Epoxide *II* (0.01 mol) in butanol was treated with *p*-bromoaniline and benzylamine (0.01 mol). The solution was heated under reflux for 3 h. The solid products after concentration and cooling were crystallized from the proper solvent. The structure of *IIIa*, *IIIb* was proved by IR spectrum, which showed absorption bands at 3350—3450 cm^{-1} (ν(OH)), and the bands for ν(C—O—C) disappeared. ^{1}H NMR spectrum of *IIIa* showed signals at δ: 3.1 (d, 1H, α-CH), 3.3 (d, 1H, β-CH), 4.5 (s, 2H, NCH_2CO), 7.8 (m, 16H, H_{arom}), 10.2 (d, 2H, NH), and 11.2 (s, 1H, OH).

N-{4-[5-(4-Chlorophenyl)-4-hydroxy-4,5-dihydro-1*H*/1-phenylpyrazolin-3-yl]phenyl}-2-(1,1,3-trioxo-1,3-dihydrobenzo[*d*]isothiazol-2-yl)acetamide (*IVa, IVb*)

A solution of epoxide *II* (0.01 mol) was refluxed in butanol with hydrazine hydrate and phenylhydrazine (0.05 mol) for 8 h, then the reaction mixture was poured into ice water. The products were separated and crystallized from the proper solvent. The structure of *IVa* and *IVb* was supported by IR spectrum, which revealed absorption bands at 1730—1740 cm^{-1} (ν(CO) of cyclic imide), 1320 and 1130 cm^{-1} ($\nu(SO_2)$), 1670 cm^{-1} (ν(CO) of amide), 1630 cm^{-1} (ν(C=N)), and 3200 cm^{-1} (ν(NH)). ^{1}H NMR spectrum of *IVb* showed signals at δ: 2.6—2.8 (d, 2H, 2CH cyclic), 4.6 (s, 2H, NCH_2CO), 5.9 (s, 1H, OH), 7.2—7.9 (m, 17 H, H_{arom}), 10.3 (s, 1H, CONH). Mass spectrum of *IVa* showed peaks at m/z (relative intensity/%): 510.5 (0.03) (M^+), 358 (0.21), 341 (2.86), 340 (4.70), 316 (11.50), 197 (3.20), 196 (25.40), 183 (100.00), 141 (7.60), 133 (31.70), 105 (36.40), 91 (27.80), 77 (43.03), 65 (13.90), 57 (21.11).

N-{4-[4-(4-Chlorophenyl)-3-aceto/bromo-2-oxo-2,3,4,5-tetrahydrofuran-5-ylcarbonyl]-phenyl}-2-(1,1,3-trioxo-1,3-dihydrobenzo[*d*]-isothiazol-2-yl)acetamide (*Va, Vb*)

The epoxide *II* was added to a cold stirred solution of sodium hydroxide (0.02 mol). Acetic acid (30 cm^3)

with ethyl acetoacetate and ethyl bromoacetate (0.015 mol) were added. The reaction mixture was stirred for 15 min, then warmed to 50 °C for 6 h. The complex was decomposed with a mixture of water and dilute HCl, then extracted with benzene and washed with aqueous $NaHCO_3$ solution (10 %) and water. The products obtained were crystallized from the proper solvent. The structure of *Va* and *Vb* was supported by IR spectrum which showed absorption bands at 1755—1765 cm^{-1} attributable to ν(CO) of furanones. Mass spectrum of *Va* showed peaks at *m/z* (relative intensity/%): 580 (0.01) (M^+), 400 (0.02), 372 (0.24), 371 (0.84), 343 (0.13), 315 (0.50), 183 (0.19), 168 (6.20), 141 (8.90), 117 (0.44), 105 (100.00), 77 (41.93), 65 (16.20).

***N*-{4-[3-(4-Chlorophenyl)-2,3-di(*p*-tolyl)-acryloyl]phenyl}-2-(1,1,3-trioxo-1,3-dihydrobenzo[*d*]isothiazol-2-yl]acetamide (*VI*)**

To a solution of epoxide *II* in dry benzene (20 cm^3), toluene (0.03 mol) and anhydrous $AlCl_3$ (0.01 mol) were added and then the reaction mixture was refluxed for 4 h. The solid that separated on cooling was crystallized from the proper solvent. The structure of *VI* was proved by IR spectrum which showed absorption bands at 1605 cm^{-1} (ν(C═C)). ^{1}H NMR spectrum of *VI* showed signals at δ: 2.1 (s, 6H, $2CH_3$), 4.4 (s, 2H, NCH_2O), 7.2—7.8 (m, 20H, H_{arom}), 9.8 (s, 1H, CONH).

***N*-{4-[3-(4-Chlorophenyl)-3-ethyl/phenyl-2-hydroxypropanoyl]phenyl}-2-(1,1,3-trioxo-1,3-dihydrobenzo[*d*]isothiazol-2-yl)acetamide (*VIIa, VIIb*)**

To a suspension of epoxide *II* (0.01 mol) in dry ether (50 cm^3) an ethereal solution of ethyl magnesium bromide or phenyl magnesium bromide (0.03 mol) was added, the reaction mixture was refluxed on a steam bath for 4 h, then decomposed with a saturated solution of ammonium chloride and extracted with ether. The solid obtained was crystallized from the proper solvent. The structure of *VIIa* and *VIIb* was proved by IR spectrum which showed absorption bands at 3450 cm^{-1} (ν(OH)). ^{1}H NMR spectrum of *VIIa* showed signals at δ: 1.3 (t, 3H, $CH_2\underline{CH_3}$), 3.1 (d, 1H, α-CH), 3.3 (d, 1H, β-CH), 4.1 (q, 2H, $\underline{CH_2}CH_3$), 4.5 (s, 2H, NCH_2CO), 7.2—7.9 (m, 12H, H_{arom}), 9.2 (s, 1H, CONH), 11.1 (s, 1H, OH).

***N*-{4-[4-(4-Chlorophenyl)-2-thioxo-2,3,4,5-tetrahydrooxazolin-4-ylcarbonyl]phenyl}-2-(1,1,3-trioxo-1,3-dihydrobenzo[*d*]isothiazol-2-yl)acetamide (*VIII*) and *N*-{4-[5-(4-Chlorophenyl)-2-oxo-2,3,5,6-tetrahydromorpholin-6-ylcarbonyl]phenyl}-2-(1,1,3-trioxo-1,3-dihydrobenzo[*d*]isothiazol-2-yl)acetamide (*IX*)**

Equimolar amounts of epoxide *II*, thiourea, and glycine (0.01 mol) were heated under reflux in DMF (20 cm^3) in the presence of catalytic amount of $AlCl_3$ (0.05 g) for 3 h. The reaction mixture was poured into water and extracted with ether. The solid obtained was crystallized from the proper solvent. The structure of *VIII* and *IX* was established by IR spectrum of *VIII* which showed absorption bands at 1260 cm^{-1} (ν(C═S)) and IR spectrum of *IX* which showed absorption bands at 1710 cm^{-1} (ν(CO) of α-lactone). Mass spectrum of *IX* showed peaks at *m/z* (relative intensity/%): 353.5 (3.80) (M^+), 414 (6.20), 400 (12.40), 372 (7.80), 371 (15.30), 343 (2.70), 315 (8.90), 196 (41.60), 168 (39.90), 140 (47.50), 119 (43.70), 104 (32.60), 91 (44.30), 76 (100.00), 65 (28.50).

***N*-{4-[3-(4-Chlorophenyl)-3-acetyl-2-hydroxypropanoyl]phenyl}-2-(1,1,3-trioxo-1,3-dihydrobenzo[*d*]isothiazol-2-yl)acetamide (*X*)**

A solution of epoxide *II* and acetic acid (30 cm^3) was refluxed for 4 h. The reaction mixture was cooled and poured into water. The separated solid was crystallized from the proper solvent. The structure of *X* was supported by IR spectrum which showed absorption bands at 3350—3450 cm^{-1} (ν(OH)). ^{1}H NMR spectrum showed signals at δ: 2.1 (s, 3H, $COCH_3$), 3.15 (d, 1H, α-CH), 3.35 (d, 1H, β-CH), 7.8 (m, 12H, H_{arom}), 9.1 (s, 1H, CONH), 11.2 (s, 1H, OH).

***N*-{4-[3-(4-Chlorophenyl)-3-acylamido-2-hydroxypropanoyl]phenyl}-2-(1,1,3-trioxo-1,3-dihydrobenzo[*d*]isothiazol-2-yl)acetamides (*XIa—XIc*)**

Equimolar amounts of epoxide *II* (0.03 mol) and BF_3 etherate (0.03 mol) as catalyst were stirred in benzonitrile, acetonitrile, and acrylonitrile (10 cm^3) as a solvent and reagent was stirred at room temperature for 5 h. The reaction mixture was poured into aqueous $NaHCO_3$ (5 %) and extracted with ether. The solid obtained was crystallized from the proper solvent. IR spectra showed absorption bands at 3150—3450 cm^{-1} (ν(NH) and ν(OH)).

N*-{4-[4-(4-Chlorophenyl)-2R′-4,5-dihydrooxazolin-5-ylcarbonyl]phenyl}-2-(1,1,3-trioxo-1,3-dihydrobenzo[*d*]isothiazol-2-yl)acetamides *XIIa—XIIc

The hydroxy amides *XIa—XIc* were heated at 210—220 °C for 6—8 h in an oil bath. The cooled pyrolyzate was dissolved in ether, filtered and dried over anhydrous sodium sulfate. The solvent was evaporated and gave the solid product which crystallized from the proper solvent. The structure of *XIIa—XIIc* was supported as follows. IR spectra showed absorption bands at 1630 cm^{-1} (ν(C═N)), 1210 cm^{-1} (ν(C—

Table 2. Physical Data of Compounds Prepared

Compound	Formula M_r	w_i(calc.)/% w_i(found)/% C	H	N	Yield/%	Solvent	M.p./°C Colour
II	$C_{24}H_{17}ClN_2O_6S$	58.0	3.4	5.6	65	B	105—107
	496.5	58.0	3.4	5.6			y
IIIa	$C_{30}H_{23}ClN_3O_6BrS$	53.9	3.4	6.3	66	B	167—169
	668.5	53.9	3.4	6.3			b
IIIb	$C_{31}H_{25}ClN_3O_6S$	61.7	4.1	7.0	64	X	139—141
	602.5	61.6	4.1	7.0			y
IVa	$C_{24}H_{19}ClN_4O_5S$	56.4	3.7	11.0	58	E	145—147
	510.5	56.3	3.6	11.0			y
IVb	$C_{30}H_{23}ClN_4O_5S$	60.5	3.9	9.4	60	E	187—189
	595.5	60.5	3.9	9.3			y
Va	$C_{28}H_{21}ClN_2O_8S$	57.9	3.6	4.8	61	B	191—193
	580.5	57.9	3.7	4.8			y
Vb	$C_{26}H_{18}ClN_2O_7BrS$	50.2	2.9	4.5	63	B	203—205
	617.5	50.1	2.8	4.5			b
VI	$C_{38}H_{29}ClN_2O_5S$	69.0	4.4	4.2	60	E	121—123
	660.5	69.0	4.4	4.2			y
VIIa	$C_{26}H_{23}ClN_2O_6S$	59.3	4.4	5.3	67	E	198—200
	526.5	59.3	4.3	5.2			y
VIIb	$C_{30}H_{23}ClN_2O_6S$	62.7	4.0	4.9	74	E	217—219
	574.5	62.7	4.1	4.9			y
VIII	$C_{25}H_{18}ClN_3O_6S_2$	54.0	3.2	7.6	75	X	161—163
	555.5	54.1	3.1	7.5			b
IX	$C_{26}H_{20}ClN_3O_7S$	56.4	3.6	7.6	63	B	183—185
	553.5	56.3	3.5	7.6			y
X	$C_{26}H_{21}ClN_2O_7S$	57.7	3.9	5.2	78	E	211—213
	540.5	57.6	3.8	5.2			y
XIa	$C_{31}H_{24}ClN_3O_7S$	60.2	3.9	6.8	67	B	173—175
	617.5	60.2	3.8	6.7			y
XIb	$C_{26}H_{22}ClN_3O_7S$	56.2	4.0	7.6	65	B	219—221
	555.5	56.2	4.1	7.5			y
XIc	$C_{27}H_{22}ClN_3O_7S$	57.1	3.9	7.4	59	B	179—181
	567.5	57.2	3.8	7.5			y
XIIa	$C_{31}H_{22}ClN_3O_6S$	66.7	3.7	7.0	61	B	134—136
	599.5	66.7	3.6	7.0			y
XIIb	$C_{26}H_{20}ClN_3O_6S$	58.0	3.7	7.8	60	B	159—161
	537.5	58.1	3.7	7.7			y
XIIc	$C_{27}H_{20}ClN_3O_6S$	59.0	3.6	7.6	59	B	151—153
	649.5	59.3	3.5	7.5			b

B = benzene; X = xylene; E = ethanol; b = brown; y = yellow.

O—C)), and the bands disappeared for ν(OH). ^{1}H NMR spectrum of *XIIc* showed signals at δ: 2.3 (d, 2H, 2CH cyclic), 4.6 (s, 2H, NCH_2CO), 6.2 (t, 1H, CH═CH), 6.9 (d, 1H, CH_2═CH—), 7.9 (m, 12H, H_{arom}), 9.9 (s, 1H, CONH). Mass spectrum of *XIIb* showed peaks at *m/z* (relative intensity/%): 537.5 (2.80) (M^+), 372 (5.70), 371 (11.50), 343 (3.70), 315 (15.50), 196 (24.00), 183 (100.00), 168 (12.20), 141 (9.60), 140 (18.50), 120 (13.30), 119 (49.10), 105 (27.30), 91 (30.20), 77 (38.50), 65 (22.60), 57 (47.40).

Antimicrobial Activity

The antimicrobial activities of all synthesized compounds were determined by using the hole plate and filter paper disc method. The tested compounds were dissolved in 10 % acetone. The concentrations chosen were 125 μg/cm^3 and 250 μg/cm^3.

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