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Synthesis, Characterizations and Antimicrobial Activity of Oxazolidinone and Imidazolidinone Derivatives

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ABSTRACT

Glycine (1) was allowed to react with substituted benzoyl chloride (2) to give substituted hippuric acid(3). Different aldehydes were treated with compound(3a-b)to get 1,3-oxazole-5-ones derivative(4a-j, 7a-j). These oxazolinones were treated with various aromatic amines to get various imidazol-5-ones derivatives (5a-j, 6a-j, 8a-j, 9a-j). Compounds characterized by elemental analyses like IR spectra, NMR spectra and GCMS. The products have been tested for their antifungal and antibacterial activity against gram (+) ve and gram (-) ve bacteria.

Keywords: Oxazolinone, Imidazol-5-Ones, Antibacterial Activity, Antifungal Activity.

I. INTRODUCTION

4-Benzylidene-2-phenyl-1, 3-oxazol-5(4H)-ones (azlactones) are molecules with important synthetic potential, several antimicrobial and technological applications[1-6]. Imidazolones[7-10] have been associated with several pharmacological activities such as antibacterial, antifungal, antiviral, anti-cancer, CNS depressant activity etc. The present antimicrobial drugs are losing their effectiveness due to the increase in microbial pathogenic resistance[7]. Imidazolinone is an important scaffold possessing a spectrum of pharmacological actions, which include anticonvulsant, antiparkinsonism and monoamino-oxidase inhibitory activities[8-13].

N.S.Hary et al. have synthesized various 1,2,4 tri substituted 5-imidazolinone derivatives. The compounds investigated for their anticonvulsant activity in chemistry. In silico metabolite and toxicity studies conducted for correctness in terms of their susceptibility to metabolism and toxicity profile [14]. Lacroix and co-workers have synthesized 2-imidazolinone-5-ones are as agrochemical Fungicides. Those compound showed control of *Puccina recondita* on wheat at the concentration of 1 µg/ml [15].

Saravanan *et al* have studied antibacterial activity of imidazolone-5-(4*H*)ones LVI. Thus, among the compounds, 1-(2-aminoethyl)-2-phenyl-4-(4-dimethylamino) benzylidene)imidazole-5-(4*H*)one,1-carboxamido-2-phenyl-4-(4-(dimethylamino) benzylidene) imidazole-5-(4*H*)one and 1-(4-(*N,N*-dimethylamino)phenyl)-2-phenyl-4-(4-(dimethylamino)benzylidene)imidazole-5-(4*H*)one were found to have a significant higher antibacterial activity than the other substituted imidazolones[16]. Reen Rathore et al. have prepared piperazinyl azlactone derivatives and tested against antibacterial and anti fungal activity[17].

The glycine (1) treated with vaious benzoyl chloride (2) in presence of sodium hydroxide soln to get acylated product (3). The required oxazolones (4a-j, 7a-j) were synthesized by th cyclization of compound (3) in presence of acetic anhydride followed by condensation with benzaldehyde. Absences of -OH and -NH_2 broad singlet peaks in ^{1}H NMR spectrum and moleculer ion peak at m\z 263.2 in mass spectrum was confirmed for its formation of compound 4a. Oxazolonenes was treated with various amines in presence of acetic acid to get imidazoline(5a-j, 6a-j, 8a-j, 9a-j).

II. METHODS AND MATERIAL

A. Experimental

All starting material, reagents and solvents are commercially available and were used without further purification. All melting points were taken in paraffin bath and are uncorrected. IR spectra were recorded on BRUKER ALPHA-E spectrometer (λ_{max} in cm^{-1}) [18]; 1H NMR were reorded on Brucker 400MHz spectrometer. Chemical shift (δ) are reported in part per million (ppm) relative to traces of $CDCl_3$ [19]. Mass spectra were recorded on Agilent Technologies 6390N. Reaction progrees was checked by TLC by keeping the plates in iodine vapor or UV lamp.

1. Preparation of substituted Hippuric acid:

A glycine(1.0 mole) was dissolved in a 1L of soldium hydroxide 10% solution and to it, benzoyl chloride (1.2 mole) was added portion wise while stirring. The reaction mixture stir until each portion of chloride has been reacted. The reaction mixture was the poured to crushed ice and acidify with concentrated HCl while stirring to pH:2.0. The precipitate of substituted hippuric acid so obtained was filtered, washed several times with cold distilled water and dry the solid. The product was recrystallized from ethylacetate and hexane. 3-methyl hippuric acid (3a). M.P 137-139^{0}C, yield: 80%. 4-Chloro hippuric acid (3b), M.P:144-146^{0}C, yield: 80%.

2. Preparation of Oxazole-5-ones derivatives [4a-j, 7a-j] :

In a round bottom flask, a mixture of hippuric acid (3a-b) (0.03mole), benzaldehyde (0.03 mole), acetic anhydride (0.03 mole) and sodium acetate (0.03 mole) was taken. The reaction mixture was heated to 50-60C in oil bath with constant shaking. As soon as the reaction mixture has been liquidify, the reaction mixture heated to 110^{0}C for 2-3 hours while stirring. Then ethanol 15gm) was added slowly while cooling and the reaction mixture was allowed to stand overnight. The resulting precipitate so obtained was filtered, washed with cold ethanol (5 gm) and cold water and crystallized from ethanol or toluene (Table1).

3. Preparation of Imidazoole-5-ones derivatives [5a-j, 6a-j, 8a-j, 9a-j] :

A mixture of Oxazole-5-one (0.0035 mole), alkyl aniline (0.007 mole) and aetic acid (15 gm) was added to 100 mL of round bottom flask. The reaction mixture was refluxed in oil batha at 120^{0}C gently for 12 hr. After completetion of reaction, the reaction mixture was poured over crushed ice and The precipitate of imidazole-5-one so obtained was filtered, washed several times with cold distilled water and dry the solid. The product was recrystallized from ethanol. The compounds are characterized by elemental analysis, IR, NMR spectrum and mass spectra.

Scheme 1

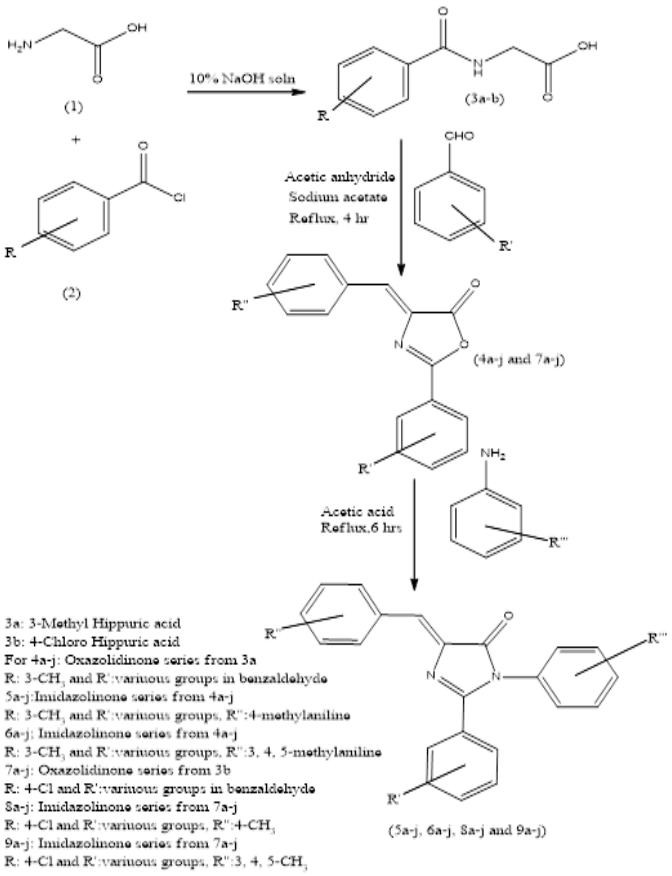

Table 1: Physical and Analitical data of the compounds

Product ID	-R''	Molecular Formula	Mol. wt (gm/m)	Yield %	MP ^0C	Carbon (%)		Hydrogen(%)		Nitrogen(%)	
						Found	Required	Found	Required	Found	Required
4A	-H	$C_{17}H_{13}NO_2$	263.29	72	132-137	77.57	77.55	5.02	4.98	5.36	5.32
4B	2-NO_2	$C_{17}H_{12}N_2O_4$	308.28	70	166-168	66.29	66.23	3.95	3.92	9.15	9.09
4C	2-Cl	$C_{17}H_{12}ClNO_2$	297.73	67	142-145	68.59	68.58	4.10	4.06	4.75	4.70
4D	4-Cl	$C_{17}H_{12}ClNO_2$	297.73	75	180-183	68.65	68.58	4.10	4.04	4.75	4.70
4E	4-OCH_3	$C_{18}H_{15}NO_3$	293.31	77	166-173	73.79	73.71	5.19	5.15	4.85	4.78
4F	3, 4-$(OCH3)_2$	$C_{19}H_{17}NO_4$	323.34	67	146-149	70.65	70.58	5.33	5.30	4.39	4.33
4G	2-OCH_3	$C_{18}H_{15}NO_3$	293.31	69	162-166	73.79	73.71	5.19	5.15	4.85	4.78
4H	4-CH_3	$C_{18}H_{15}NO_2$	277.31	68	157-161	77.99	77.96	5.49	5.45	5.16	5.05
4I	3-Br	$C_{17}H_{12}BrNO_2$	342.18	72	147-150	59.75	59.67	3.56	3.53	4.11	4.09
4J	3,4,5$(OCH_3)_3$	$C_{20}H_{19}NO_5$	353.36	17	133-136	68.05	67.98	5.46	5.42	3.99	3.96
5A	-H	$C_{24}H_{20}N_2O$	352.42	68	208-212	81.82	81.79	5.75	5.72	7.96	7.95
5B	2-NO_2	$C_{24}H_{19}N_3O_3$	397.42	72	165-169	72.56	72.53	4.85	4.82	10.59	10.57
5C	2-Cl	$C_{24}H_{19}ClN_2O$	386.87	74	182-186	74.55	74.51	4.98	4.95	7.26	7.24
5D	4-Cl	$C_{24}H_{19}ClN_2O$	386.87	71	188-192	74.55	74.51	4.98	4.95	7.26	7.24
5E	4-OCH_3	$C_{25}H_{22}N_2O_2$	382.45	67	168-172	78.56	78.51	5.85	5.80	7.36	7.32
5F	3, 4-$(OCH3)_2$	$C_{26}H_{24}N_2O_3$	412.48	69	194-197	75.73	75.71	5.86	5.86	6.81	6.79
5G	2-OCH_3	$C_{25}H_{22}N_2O_2$	382.45	73	186-190	78.56	78.51	5.85	5.80	7.36	7.32
5H	4-CH_3	$C_{25}H_{22}N_2O$	366.45	68	188-191	81.95	81.94	6.08	6.05	7.66	7.64
5I	3-Br	$C_{24}H_{19}BrN_2O$	431.32	69	168-172	66.85	66.83	4.46	4.44	6.51	6.49
5J	3,4,5$(OCH_3)_3$	$C_{27}H_{26}N_2O_4$	442.50	74	212-217	73.32	73.28	5.93	5.92	6.36	6.33
6A	-H	$C_{26}H_{24}N_2O$	380.48	68	128-132	82.09	82.07	6.39	6.36	7.39	7.36
6B	2-NO_2	$C_{28}H_{31}N_3O_3$	425.47	72	163-167	73.41	73.39	5.46	5.45	9.94	9.88
6C	2-Cl	$C_{26}H_{23}ClN_2O$	414.92	73	154-157	75.29	75.26	5.62	5.59	6.78	6.75
6D	4-Cl	$C_{26}H_{23}ClN_3O$	414.92	69	146-150	75.29	75.26	5.62	5.59	6.78	6.75
6E	4-OCH_3	$C_{27}H_{26}N_2O_2$	410.50	73	153-157	79.02	79.00	6.42	6.38	6.85	6.82
6F	3, 4-$(OCH3)_2$	$C_{28}H_{28}N_2O_3$	440.53	71	166-170	76.36	76.34	6.45	6.41	6.39	6.36
6G	2-OCH_3	$C_{27}H_{26}N_2O_2$	410.50	74	156-159	79.02	79.00	6.39	6.36	6.85	6.82
6H	4-CH_3	$C_{27}H_{26}N_2O$	394.50	71	158-161	82.22	82.20	6.69	6.64	7.12	7.10
6I	3-Br	$C_{26}H_{23}BrN_2O$	459.37	69	180-185	67.99	67.98	5.09	5.05	6.12	6.10
6J	3,4,5$(OCH_3)_3$	$C_{29}H_{30}N_2O_4$	470.55	72	263-267	74.03	74.02	6.46	6.43	5.98	5.94
7A	-H	$C_{16}H_{10}ClNO_2$	283.70	76	180-183	67.76	67.74	3.59	3.55	4.96	4.94
7B	2-NO_2	$C_{19}H_9ClN_2O_4$	328.70	74	228-233	58.49	58.46	2.79	2.76	5.54	8.52
7C	2-Cl	$C_{16}H_9Cl_2NO_2$	318.15	73	226-230	60.44	60.40	2.86	2.85	4.44	4.40
7D	4-Cl	$C_{16}H_{10}ClNO_2$	318.15	67	226-230	60.44	60.40	2.86	2.85	4.44	4.40
7E	4-OCH_3	$C_{17}H_{12}ClNO_3$	313.73	69	178-182	65.09	65.08	3.89	3.86	4.49	4.46
7F	3, 4-$(OCH3)_2$	$C_{18}H_{14}ClNO_4$	343.76	71	205-208	62.92	62.89	4.12	4.10	4.09	4.07
7G	2-OCH_3	$C_{17}H_{12}ClNO_3$	313.73	73	178-182	65.12	65.08	3.89	3.86	4.49	4.46
7H	4-CH_3	$C_{17}H_{12}ClNO_2$	297.73	69	201-205	68.62	68.58	4.09	4.06	4.72	4.70
7I	3-Br	$C_{16}H_9BrClNO_2$	362.60	67	181-185	53.05	53.00	2.53	2.50	3.89	3.86
7J	3,4,5$(OCH_3)_3$	$C_{19}H_{16}ClNO_5$	373.78	70	186-190	61.06	61.05	4.33	4.31	3.76	3.75
8A	-H	$C_{23}H_{17}ClN_2O$	372.84	75	197-202	74.12	74.09	4.62	4.60	7.53	7.51
8B	2-NO_2	$C_{23}H_{16}ClN_3O_3$	417.84	73	161-166	66.13	66.11	3.89	3.86	10.08	10.06
8C	2-Cl	$C_{23}H_{16}Cl_2N_2O$	407.29	71	175-179	67.85	67.83	3.98	3.96	6.89	6.88
8D	4-Cl	$C_{23}H_{16}Cl_2N_2O$	407.29	69	234-238	67.85	67.83	3.98	3.96	6.89	6.88
8E	4-OCH_3	$C_{24}H_{19}ClN_2O_2$	402.87	72	198-202	71.56	71.55	4.76	4.75	6.96	6.95
8F	3, 4-$(OCH3)_2$	$C_{25}H_{21}ClN_2O_3$	432.89	67	170-175	69.39	69.36	4.89	4.89	6.52	6.47

8G	2-OCH_3	$C_{24}H_{19}ClN_2O_2$	402.87	72	202-206	71.56	71.55	4.76	4.75	6.98	6.95
8H	4-CH_3	$C_{24}H_{19}ClN_2O$	386.87	76	19-195	74.56	74.51	4.96	4.95	7.26	7.24
8I	3-Br	$C_{23}H_{16}BrClN_2O$	451.74	69	177-181	61.19	61.15	3.62	3.57	6.24	6.20
8J	3,4,5$(OCH_3)_3$	$C_{26}H_{23}ClN_2O_4$	462.92	71	233-238	67.49	67.46	5.03	5.01	6.07	6.05
9A	-H	$C_{25}H_{21}ClN_2O$	400.90	75	142-145	74.92	74.90	5.32	5.28	7.01	6.99
9B	2-NO_2	$C_{25}H_{20}ClN_3O_3$	445.89	73	181-185	67.36	67.34	4.55	4.52	9.42	9.42
9C	2-Cl	$C_{25}H_{20}Cl_2N_2O$	435.34	69	195-200	68.99	68.97	4.67	4.63	6.46	6.43
9D	4-Cl	$C_{25}H_{20}Cl_2N_2O$	435.34	71	183-185	68.99	68.97	4.67	4.63	6.46	6.43
9E	4-OCH_3	$C_{26}H_{23}ClN_2O_2$	430.92	68	166-170	72.49	72.47	5.42	5.38	5.53	6.50
9F	3, 4-$(OCH3)_2$	$C_{27}H_{25}ClN_2O_3$	460.95	69	210-214	70.39	70.35	5.49	5.47	6.12	6.08
9G	2-OCH_3	$C_{26}H_{23}ClN_2O_2$	430.92	73	228-232	72.52	72.47	5.42	5.38	6.52	6.50
9H	4-CH_3	$C_{26}H_{23}ClN_2O$	414.92	72	191-195	75.29	75.26	5.62	5.59	6.77	6.75
9I	3-Br	$C_{25}H_{20}BrClN_2O$	479.79	73	176-180	62.62	62.58	4.25	4.20	5.86	5.84
9J	3,4,5$(OCH_3)_3$	$C_{28}H_{27}ClN_2O_4$	490.97	71	211-216	68.53	68.50	5.60	5.54	5.74	5.71

4. A:4-benzylidene-2-(3-methylphenyl)-1,3-oxazol-5(4H)-one

1H NMR (CDCl3) δ(ppm): 2.47ppm (s, 3H, -CH3), 7.25-8.22ppm (m, 10H, Ar-CH and C=CH), 13C NMR(CDCl3) δ(ppm): 21.37, 125.47, 125.67,128.61, 128.82, 128.87, 128.93, 131.17, 131.57, 132.46, 133.36, 133.58, 134.29, 138.85, 163.74, 167.73ppm. IR: 3020 (-CH stretch, aromatic), 1800 (>C=O stretch, cyclic ring), 1650 (>C=N stretch, oxazole ring), 1580 (>C=C< aromatic), 1320(-C-O stretching);GCMS: Fragmentation of mass spectra m/z : 263.2(M^+), 207, 119.2, 91.1, 65.1 and 39.1

5. A:2-(3-methylphenyl)-4-(benzylidene)-1-(4-methylphenyl)-4H-imidazol-5-one

1H NMR (CDCl3) δ(ppm): 2.25ppm (s, 3H, -CH3), 2.31ppm (s, 3H, -CH3), 6.97-8.22ppm(m,14H, Ar-CH and C=CH), 13C NMR(CDCl3) δ(ppm): 21.25, 21.41,126.42, 127.13, 128.05, 128.80, 129.03, 129.89, 130.40, 132.11, 132.19, 132.60, 134.45, 138.25, 138.34, 138.69, 160.96 and 170.80. IR: 3000 (-CH stretch, aromatic), 1720 (>C=O stretch, cyclic ring), 1620 (>C=N stretch, imidazole ring), 1510 (>C=C< aromatic), 1250(-C-N tertiary amine);GCMS: Fragmentation of mass spectra m/z : 352.2 (M^+), 261.1, 208.1, 165.1, 119.1, 91.1, 65.1, 39.1

6. **A: 2-(3-methylphenyl)-4-(benzylidene)-1-(3,4,5-trimethylphenyl)-4H-imidazol-5-one**

1H NMR (CDCl3) δ(ppm): 1.98 (s, 6H, -CH3), 2.22ppm (s, 3H, -CH3), 2.25ppm (s, 3H, -CH3), 6.88ppm - 8.25ppm (m,12H, Ar-CH and C=CH). 13C NMR(CDCl3) δ(ppm): 18.12, 18.39, 21.2, 21.47, 124.97, 128.34, 128.75, 128.84, 128.92, 129.07, 129.63, 130.41, 130.67, 132.55, 132.62, 134.48, 135.48, 135.34, 135.97, 138.35, 138.62, 139.22, 160.74 and 170.75;IR: 2880 (-CH stretch, aromatic), 1720 (>C=O stretch, cyclic ring), 1620 (>C=N stretch, imidazole ring), 1500 (>C=C< aromatic), 1250(-C-N tertiary amine);GCMS: Fragmentation of mass spectra m/z: 380.3(M^+), 281.1, 236.2, 207.1, 177, 147.1, 119.1, 91.1, 65.1, 39.1

7. **A: 4-benzylidene-2-(4-chlorophenyl)-1,3-oxazol-5(4H)-one**

1H NMR (CDCl3): 7.20-8.11ppm(m, 10H, Ar-CH and C=CH). 13C NMR(CDCl3): 124.09, 128.98, 129.43, 129.60, 131.42, 132.35, 132.54, 133.03, 133.42, 139.83, 162.68 and 167.38 IR: 3050 (-CH stretch, aromatic), 1800 (>C=O stretch, cyclic ring), 1640 (>C=N stretch, oxazole ring), 1500 (>C=C< aromatic), 1310(-C-O stretching), 750 (-C-Cl).GCMS: Fragmentation of mass spectra m/z: 283.1(M^+), 207.1,139.1, 111.1, 75.1, 39.1

8. A:2-(4-chlorophenyl)-4-(benzylidene)-1-(4-methylphenyl)-4H-imidazol-5-one

1H NMR (CDCl3) (δ ppm): 2.32ppm (s, 3H, -CH3), 6.99-8.19ppm(m, 14H, Ar-CH and C=CH). 13C NMR(CDCl3) (δ ppm): 21.27, 119.78, 127.13, 127.34, 128.71, 128.84, 129.64, 129.80, 130.28, 130.55, 130.61, 131.84, 132.66, 134.33, 137.70, 138.44, 138.72, 159.57 and 170.61 IR: 2922 (-CH stretch, aromatic), 1720

(>C=O stretch, cyclic ring), 1638 (>C=N stretch, imidazole ring), 1514 (>C=C< aromatic), 1293(-C-N tertiary amine), 757 (-C-Cl).GCMS: Fragmentation of mass spectra m/z: 372.2(M^+), 281.1, 207.1, 177.0, 139.0, 91.1, 65.1, 39.1

9. A:2-(4-chlorophenyl)-4-(benzylidene)-1-(3,4,5-trimethylphenyl)-4H-imidazol-5-one

1H NMR (CDCl3) (δ ppm): 1.97ppm (s, 6H, -CH3), 2.64ppm (s, 3H, -CH3), 6.90-8.23ppm(m, 12H, Ar-CH and C=CH). 13C NMR(CDCl3) (δ ppm): 18.08, 18.32, 21.21, 127.61, 128.87, 128.96, 129.34, 129.38, 129.83, 130.35, 130.62, 132.68, 134.34, 135.94, 138.05, 138.35, 139.54, 159.37 and 170.51 IR: 2880 (-CH stretch, aromatic), 1720 (>C=O stretch, cyclic ring), 1640 (>C=N stretch, imidazole ring), 1510 (>C=C< aromatic), 1300(-C-N tertiary amine), 780 (-C-Cl).GCMS: Fragmentation of mass spectra m/z: 400.3 (M+), 350.2, 309.2, 282.2, 256.2, 207.1, 179.1, 139.1, 91.2, 41.2

III. BIOLOGICAL ACTIVITY:

Antibacterial and Antifungal Activity

The synthesized compounds were screened for threir *in-vitro* antimicrobial activity against *Escherechia.coli* (Gram negative), *Pseudomonas aeruginosa (*Gram negative*), Staphylococcus aureus(*Gram positive*), Streptococcus pyogenes*(Gram positive) and antifungal activity against *Candida albicans, Aspergillus niger* and *Aspergillus clavatus* by measuring in MBC and in MFC method in µg/ml. The synthesized compounds were compared with standard antibecterial drugs Gentamycin, Ampicilin, Chloramphenicol, Ciprofloxacin, Norfloxacin and antifungal drugs Nystatin and Greseofulvin.Antibacterial and antifungal activity was carried out by broth dilution method at concentrations of 1000, 500, 250, 200, 125, 100, 62.5 µg/ml respectively[20] (Table 2).

Table 2 : Antibacterial and Antifungal Activities

Product Code	Minimal bactericidal concentration (MBC) in µg/ml				Minimal fungicidal concentration (MFC) in µg/ml		
	Gram negative bacteria		Gram positive bacteria		Fungus		
	E.coli	*P.aeruginosa*	*S.aureus*	*S.pyogenus*	*C.albicans*	*A.nigar*	*A.clavatus*
	MTCC 443	MTCC 1688	MTCC 96	MTCC 442	MTCC 227	MTCC 282	MTCC 1323
4B	250	200	500	500	200	>1000	>1000
4C	200	250	200	200	500	>1000	>1000
4J	62.5	200	500	500	200	500	500
5D	200	200	500	500	500	1000	1000
5G	100	200	250	250	250	>1000	>1000
5H	250	250	100	200	1000	500	1000
5I	250	250	200	250	100	500	500
6A	200	200	500	250	200	1000	1000
6E	200	200	500	500	1000	250	250
6F	100	62.5	250	250	1000	200	200
6J	200	200	250	500	250	>1000	>1000
7A	125	200	62.5	100	250	>1000	>1000
7C	250	250	62.5	200	250	>1000	>1000
7F	200	100	100	100	1000	200	250
8E	200	500	200	200	500	>1000	>1000
8F	250	500	200	200	500	>1000	>1000
8H	250	500	100	100	250	>1000	>1000
8J	250	100	250	250	500	200	500
9B	100	200	250	500	500	>1000	>1000
9D	250	200	200	500	1000	>1000	>1000

9G	100	200	250	500	1000	500	500
9I	250	250	250	250	1000	500	500
Gentamycin	0.05	1	0.25	0.5	--	--	--
Ampicilin	100	--	250	100	--	--	--
Chloramphenicol	50	50	50	50	--	--	--
Ciprofloxacin	25	25	50	50	--	--	--
Norfloxacin	10	10	10	10	--	--	---
Nystatin	--	--	--	--	100	100	100
Greseofulvin	--	--	--	--	500	100	100

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V. REFERENCES

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