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Synthesis and Antimicrobial Study of Several New 3-Aryl –5(9'-Phenanthryl)-Azoles

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Abstract. Phenanthryl chalcones (1) reacts with hydrazine hydrate and phenyl hydrazine to produce 2-pyrazloline derivatives (3) and (4) respectively. Acetylation of (3) gave (5) which can obtained also from compounds (1). Oxidation of compound (1) afforded oxirane derivatives (6). Isoxazolines (7) were also prepared. Compounds (7) reacted with N-bromosuccinimide and yielded isoxazoles (8). These isoxazoles have been prepared from chalcones (1) and oxirane derivatives (6). Reaction of (7) with phosphours pentasulphide leads to the formation of 2-isothiazoline derivatives (9). The biological activity of the prepared compounds were also studied.

Introduction

A wide variety of pharmacological properties have encountered with di-substituted pyrazoline and isoxazoline derivatives including antiflammator, antiallergitic and antidiabetic activity [1-4].

Experimental

All melting points are uncorrected and were determined in capillary tube. IR spectra were recorded in potassium bromide on Perkin-Elmer 380 and 783 spectrometers. The ^{1}HNMR spectra were recorded on Varian Ex100 or FTNMR. Jummex 40 spectrometers using TMS as an internal reference in $CDCl_3$. The elemental analyses were performed on a Heraus CHN analyser. The UV spectra were obtained with UV-Vis-spectrophoto-meter (160 A-UV) in chloroform. Physical and analytical properties of the prepared compounds are shown in Table 1, while the spectral data are depicted in Tables 2 and 3.

3-Aryl-5(9'-phenanthryl)-2-propen-1-ones (**1a-e**) were synthnsized according to the literature procedure [5].

1H- 3-Aryl-5-(9'-phenanthryl)-2-pyrazolines (3a-b)

A mixture of**(Ia,b)** (0.01 mol) and hydrazine hydrate (0.02 mol, 98%) in (15 ml) ethanol was heated under reflux for 4 hr. The reaction mixture was then cooled, poured into ice-cold water, and the product was collected by filtration and recrystallized from the proper solvent (Tables 1 and 3).

Table 1. Analytical and physical data of compounds (3-9)

Compd. No.	M.p.°C Solvent	Yield %	Mol. Formula M.W	% Analysis C	Calcd/Found H	N
3a	194	82	$C_{23}H_{18}N_2$	85.71	5.59	8.69
	AcOH		322	85.68	5.65	8.36
3b	150	83	$C_{23}H_{17}ClN_2$	77.41	4.76	7.85
	AcOH		356.5	77.90	4.47	7.49
4a	232	89	$C_{29}H_{22}N_2$	87.43	5.52	7.03
	AcOH		398	87.84	5.67	7.49
4b	195	87	$C_{29}H_{21}ClN_2$	80.46	4.85	6.47
	EtOH		432.5	80.29	4.86	6.93
5a	173	84.5	$C_{25}H_{20}N_2O$	82.41	5.49	7.69
	AcOH		364	82.89	5.08	7.54
5b	110	85	$C_{25}H_{19}ClN_2O$	75.28	4.76	7.02
	EtOH		398.5	75.27	4.50	7.12
6a	120	45	$C_{24}H_{18}O_2$	85.20	5.32	
	EtOH		338	85.03	5.30	
6b	210	48	$C_{23}H_{15}BrO_2$	68.50	3.72	
	EtOH		402.9	68.18	3.70	
6c	123	53	$C_{29}H_{20}O_2$	87.00	5.00	
	MeOH		400	87.30	5.20	
7a	300	81	$C_{23}H_{17}NO$	85.44	5.26	4.33
	EtOH		323			
7b	118	79	$C_{24}H_{19}NO$	85.45	5.63	4.15
	EtOH		337	85.89	5.44	4.14
7c	200	88	$C_{23}H_{16}ClNO$	77.20	4.47	3.91
	EtOH		375.5	77.23	4.02	3.73
7d	158	88	$C_{23}H_{16}BrNO$	68.67	3.98	3.48
	MeOH		401.9	68.60	3.56	3.40
7e	100	86	$C_{29}H_{21}NO$	87.21	5.26	3.50
	EtOH+H_2O		399	87.05	5.64	3.38
8a	110	88	$C_{24}H_{17}NO$	85.97	5.07	4.17
	EtOH		335	85.90	5.67	4.11
8b	218	89	$C_{23}H_{14}BrNO$	69.01	3.50	3.50
	Benzene		399.9	69.27	3.23	3.38
8c	100	88	$C_{29}H_{19}NO$	87.65	4.78	3.52
	MeOH-H_2O		397	87.60	4.73	3.50
9a	143	80	$C_{23}H_{17}NS$	81.41	5.01	4.12
	MeOH		339	81.43	5.03	4.22
9b	100	88	$C_{24}H_{19}NS$	81.58	5.38	3.96
	EtOH		353	81.52	5.87	3.88
9c	140	83	$C_{23}H_{16}ClNS$	73.89	4.28	3.74
	EtOH		373.5	73.84	4.27	3.80
9d	115	85	$C_{23}H_{16}BrNS$	66.04	3.82	3.35
	CH_2Cl_2-MeOH		417.9	66.00	3.85	3.25

N-Phenyl-3-aryl-5 (9'-phenanthryl)-2-pyrazolines (4a,b)

A solution of chalcone **(Ia,b)** (0.01 mol) and phenylhydrazine (0.015 mol) in ethanol (20 ml) was heated under reflux for 5 hr. On cooling the resulting solid was washed with water and crystallized from acetic acid (Tables 1 and 3).

N-Acetyl-3-aryl-5(9'-phenanthryl)-2-pyrazolines (5a,b)

Method A:

A mixture of compound **(1a,b)** (0.01 mol) in acetic acid (20 ml) was heated under reflux with hydrazine hydrate (0.02 mol, 98%) for 5 hr. The reaction mixture was cooled and poured on to crushed ice, the precipitate was filtered , washed with water and recrystallized from the proper solvent.

Method B : from pyrazolines (3)

A solution of compound **(3a,b)** (0.01 mol) in acetic anhydride (20 ml) was heated under reflux for 3 hr. The reaction mixture was cooled and poured on to crushed ice, the precipitate was filtered, washed with water and recrystallized from the proper solvent.(Tables 1 and 3).

2-Aroyl-3(9'-phenanthryl) oxiranes (6a-c)

A solution of compounds **(1b,d,e)** (0.01 mol) in dioxane-methanol mixture (4:1) (50 ml) was treated with aqueous sodium hydroxide solution (10 ml, 8%), followed by hydrogen peroxide (8ml, 30%). The reaction mixture was stirred and brought to boiling during 1hr., then heated under reflux on water bath for 1 hr. cooled and poured on ice, then acidified with hydrochloric acid. The solid product was filtered and washed with water. The product obtained was dried and recrystallized from the suitable solvent. (Tables 2 and 3).

3-Aryl-5(9'-phenanthryl)-2-oxa-isoxazolines (7a-e):

A mixture of compounds **(1a-e)** (0.01 mol) and hydroxyl amine hydrochloride (0.015 mol) in (30 ml) ethanol was heated under reflux for 3 hr. The reaction mixture was cooled and poured on to crushed ice. The precipitated product was filtered and recrystallized from appropriate solvent. (Tables 2 and 3).

3-Aryl-5(9'-phenanthryl)-oxazoles (8a-d)

Method A: From 2-Isoxazolines (7)

To a solution of 2-isoxazolines (**7b,d,e**) (0.01 mol) in carbon tetrachloride (40 ml); N-bromosuccinimide (0.01 mol) and trace of $\alpha-\alpha'$-azoisobutric nitrile were added and the mixture heated under reflux for 3-5 hr, the bright red color initiatly developed disappeared toward the end of the reaction. The reaction mixture was cooled and fused potassium acetate (8.0 g) and glacial acetic acid (3ml) was added, and the mixture was heated under reflux for 1hr. The resulting mixture was cooled and poured on crushed ice containing (6.0 g) sodium hydroxide. The organic layer was separated and the aqueous

layer extracted twice with ether. The combined organic was dried and solvent removed , residue obtained were crystallized from suitable solvent (Tables 2 and 3).

Method B: From chalcones (1b,d,e)

To a solution of hydroxyl amine hydrochloride (0.015 mol) in ethanol (20 ml) was added sodium acetate (2.5 g) dissolved in hot acetic acid (10 ml). The solution was added to a solution of **(1b,d,e)** (0.1 mol) in ethanol (30 ml). The mixture was heated under reflux for 2 hr. Then diluted with (250 ml) water and the solution neutralized with dilute NaOH to give the isoxazole derivatives (Tables 2 and 3).

Method C: From oxirane derivatives (6)

A mixture of compounds (**6a-c**) (0.01 mol), hydroxylamine hydro-chloride (0.02 mol) in pyridine (20 ml) was heated under reflux for 6 hr. The reaction mixture was poured in ice and the precipitated solid was filtered and recrystallized from proper solvent (Tables 2 and 3).

3-Aryl-5 (9'-phenanthryl)-2-isothiazolines (9a-d)

A solution of the appropriate isoxazolines **(7a-d)** (0.01 mol) in dry pyridine (15 ml) and phosphours pentasulphide (0.12 mol). The reaction mixture was heated under reflux for 2 hr. After cooling, it was diluted with diluted HCl, the product was filtered and recrystallized from the proper solvent. (Tables 2 and 3).

Result and Discussion

1-Aryl-3 (9`-phenanthryl)2-propen-1-ones (chalcones) **(1a,b)** were condensed with hydrazine hydrate and phenyl hydrazine to produce in a good yield 1H-3- aryl-(9-phenanthryl)-2-pyrazolines **(3a,b)** and N-phenyl-3-aryl-5-(9'phenanthryl)-2-pyrazolines **(4a,b)** respectively(Scheme 1). The structures of synthesized compounds were mainly confirmed by spectroscopic methods (Tables 1 and 3).

Thus, IR spectra of **(3a,b)** exhibited two bands at 3400-3450 cm^{-1} due to NH, while the IR spectra of**(3a,b)** and N-phenyl derivatives **(4a,b)** revealed absorption bands at 1580-1600 cm^{-1} due to C=N group.

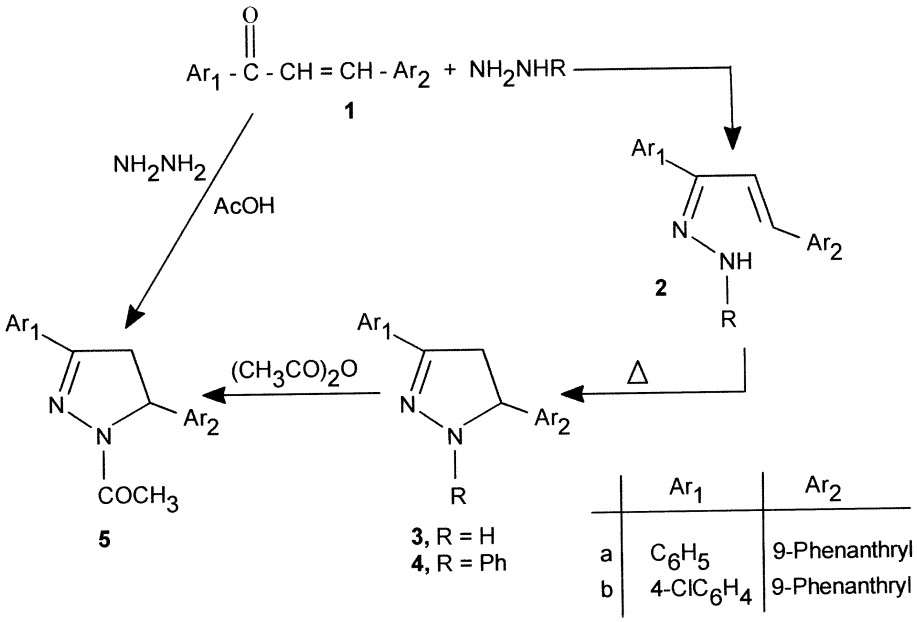
The ^{1}H NMR spectrum of compounds **(3a,b)** showed characteristic signals corresponding to protons at C4 and C5 of 2-pyrazoline ring, they form a typical ABX system confirming the nonequivalence of protons at C4 (Table 1). The mass spectrum of **(3a)** showed a molecular ion at m/z = 322 calculated for $C_{23}H_{18}N_2$.The electronic spectra of 2-pyrazolines **(3a,b)** absorb in the regions 310-275 and 238-221 nm.

The mechanism of the formation of the above 2-pyrazolines may proceed via formation of α,β-unsaturated ketone phenyl hydrazones **(2)** which cyclize through a steroselective enamine-imine tautomerism step [5].

The structure of the pyrazolines **(3a,b)** were further substantiated by their acetylation with acetic anhydride to the corresponding N-acetyl- derivatives **(5a,b)** .

However, these compounds **(5a,b)** could be prepared from **(1a,b)** (Scheme 1) [5-8]. The structure of these compounds **(5a,b)** were established by elemental analysis and spectral data (Tables 1and 3) .

IR spectra of compounds **(5a,b)** show absorption bands at region 1670-1680cm^{-1} due to C=O of the acetyl group. The ^{1}HNMR spectra gave further support to the structures of these acetylated pyrazolilnes **(5a,b)**. Thus, the latter spectra show an ABX pattern in which each of the a,b and c protons is represented by a pair of doublet, with J_{ab}=18Hz, J_{ac}=6Hz and J_{bc}=10-11 Hz. The N-acetyl protons of each compounds are characterized by a 3H singlet at δ 2.50. The electronic spectra of the acetylated pyrazolines show that they absorb at longer wavelength than the corresponding pyrazolines **(3a,b)**, since The N-acetyl derivatives **(5a,b)** show two major maxima in the regions 285-275 and 315-330 nm.



Scheme 1

Moreover, oxidation of **(1b,d,e)** with hydrogen peroxide in dioxan-methanol mixture afforded oxirane derivatives **(6a-c)** in good yield as expected [7,9].

The IR spectra of oxirane derivatives **(6a-c)** showed band at 1670-1680 cm^{-1} due to C=O group, while the ^{1}HNMR spectrum of compound **(6a)** in $CDCl_3$ showed signals at δ 2.4(s, 1H, CH_3), δ 4.12 (d, 1H, H-a), δ 4.34 (d, 1H, H-b), δ 7.2-8.8 (m, 13H, ArH's) , (Table 2).

The mass spectrum of compound **(6a)** showed a molecular ion at m/z = 338 calculated for $C_{24}H_{18}O_2$.

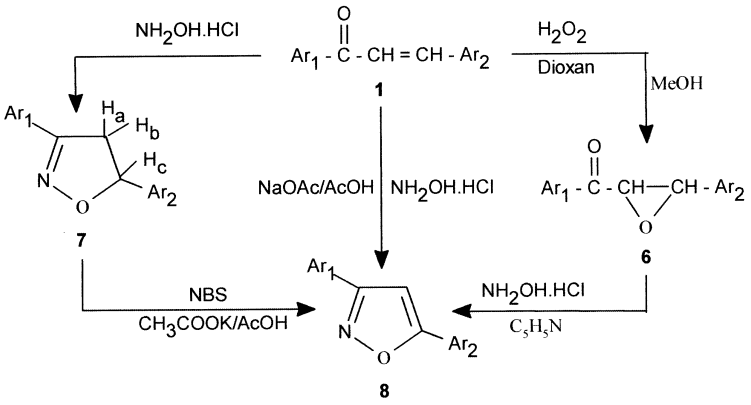
It is known that β-unsaturated ketones react with hydroxylamine to give 2-isoxazoline derivatives. Although chalcones have been described to undergo this reaction, very little has been reported on phenanthryl and biphenyl chalcones. In the present work, it is found that when **(1a-e)** are allowed to react with hydroxylamine hydrochloride in ethanol solution, the 2-isoxazoline derivatives **(7a-e)** were obtained. The structures of these new compounds were confirmed by combination of elemental and spectra data (Tables 1 and 3).

IR spectrum of compound **(7a-e)** showed band at 1580-1600 and 1610-1640 due to C=N and C=C.

Further insight concerning the structure of 2-isoxazolines **(7a)** can be gleaned from the consideration of their ^{1}HNMR spectral data (Table 2). The aromatic protons of 3,5-substituents in **(7a-e)** show multiplet signals in the range 6.8-8.8 ppm. The three protons at C-4 and C-5 of the 2-iso-xazolines are represented by typical (ABX) system. Each of the H_a, H_b and H_c is represented by a double doublet. The structure of **(7a)** was also confirmed by its mass spectrum which showed a molecular ion peak at m/z=323 calculated for $C_{23}H_{17}NO$.

It was found that N-bromosuccinimide is suitable for bromination of 2-isoxazolines **(7)** and subsequent dehydrobromination to isoxazoles occur ;usually the intermediate bromo derivatives has not been isolated although the reaction mixture was treated directly with weak bases (potassium acetate) [9].

The isoxazoles **(8a-c)** also can be obtained directly by the reaction of chalcones **(1b,d,e)** and oxiranes **(6a-c)** derivatives with hydroxylamine hydrochloride (Scheme 2) in acidic and basic media respectively.The structures of compounds **(8a-c)** have been confirmed by elemental analysis, and their spectral data (IR and ^{1}HNMR) data (Tables 1 and 3) (Scheme 2).

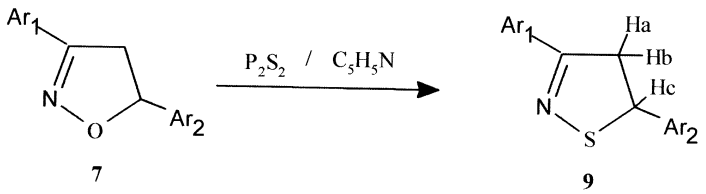


	Ar_1	Ar_2
1,7 a	C_6H_5	9-Phenanthryl
b	4-$CH_3C_6H_4$	9-Phenanthryl
c	4-ClC_6H_4	9-Phenanthryl
d	4-BrC_6H_4	9-Phenanthryl
d	4-PhC_6H_4	9-Phenanthryl

	Ar_1	Ar_2
6,8 a	4-$CH_3C_6H_4$	9-Phenanthryl
b	4-BrC_6H_4	9-Phenanthryl
c	4-PhC_6H_4	9-Phenanthryl

Scheme 2

In the present work, we report the synthesis of the isothiazolines **(9a-d)** (Scheme 3) in one step reaction via isoxazolines **(7)** and in good yield**.** Reaction of phosphours pentasulphide in pyridine bring about replacement of ring oxygen by sulphur atom [10].



	Ar_1	Ar_2
7,9 a	C_6H_5	9-Phenanthryl
b	4-$CH_3C_6H_4$	9-Phenanthryl
c	4-ClC_6H_4	9-Phenanthryl
d	4-BrC_6H_4	9-Phenanthryl

Scheme 3

The IR spectra of compounds **(9a-d)** show absorption band at 820-830cm^{-1} due to C-S function and at 915-920 cm^{-1} for S-N group. The structures of compounds **(9a-d)** were further supported by their ^{1}HNMR spectra (Table 2). The mass spectrum of compound **(9b)** showed molecular ion at m/z=353 corresponding to formula $C_{23}H_{19}NS$.

Table 2. IR and ^{1}H NMR spectral data of compounds (3-5)

Compd. No.	IR υ (cm^{-1})	^{1}H NMR (δ ppm in CD Cl_3)	J = H_z
3a	3400 (NH) , 1590, 1600 (C=N δ C=C).	3.09 (dd, 1H, H-a); 3.8 (dd, 1H, H-b); 6.4 (d-d, 1H, H-c); 7.2-8.4 (m, 14H, ArH's); 8.6 (s, 1H, NH).	Jab = 17 Jbc = 10 Jac = 8
3a	3450 (br.NH); 1580, 1610 (C=N, C=C).	3.3 (dd, 1H, H-a), 3.8 (dd, 1H, H-b); 6.4 (dd, 1H, H-c); 7.4-8.6 (m, 13H, ArH's); 8.4 (br. s, 1H, NH).	Jab = 17 Jbc = 10 Jac = 8
4a	1600, 1580 (C=N, C=C).	3.2(dd, 1H, H-a); 3.8 (dd, 1H, H-b); 5.9 (dd, 1H, H-c); 7.2-8.9 (m, 19H, ArH's).	Jab = 17 Jbc = 12 Jac = 7
4b	1600, 1850 (C=N, C=C).	3.39(dd, 1H, H-a); 3.8 (dd, 1H, H-b); 5.7 (dd, 1H, H-c); 6.8-8.5 (m, 18H, ArH's).	Jab = 17 Jbc = 12 Jac = 8
5a	1670 (C=O); 1540, 1600 (C=N δ C=C).	2.5 (s, 3H, CH_3); 3.3(dd, 1H, H-a); 3.75 (dd, H, H-b); 5.85 (dd, 1H, H-c); 6.8-7.9 (m, 14H, ArH's).	Jab = 18 Jbc = 11 Jac = 6
5b	1680 (C=O); 1560, 1590 (C=N, C=C).	2.5 (s, 3H, CH_3); 3.3(dd, 1H, H-a); 3.75 (dd, 1H, H-b); 5.85 (dd, 1H, H-c); 6.8-7.9 (m, 14H, ArH's).	Jab = 18 Jbc = 10 Jac = 6

Table 3. IR and ^{1}H-NMR spectral data of compounds (6-9)

Compd. No.	IR υ (cm^{-1})	^{1}H-NMR (δ ppm in CD Cl_3)	J = H_z
6a	1680 (C=O); 1580, 1600 (C=N δ C=C)	2.4 (s, 3H, CH_3); 4.1(d, 1H, H-a); 4.3 (d, 1H, H-b); 7.2-8.8 (m, 13H, ArH's).	Jab = 2.5
6b	1680 (C=O); 1610, 1580 (C=N δ C=C)	4.1 (d, 1H, H-a); 4.3 (d, 1H, H-b); 7.3-8.8 (m, 13H, ArH's).	Jab = 2.2
6c	1680 (C=O); 1610, 1580 (C=N δ C=C)	4.2 (d, 1H, H-a); 4.4 (d, H, H-b); 7.2-8.8 (m, 18H, ArH's).	Jab = 2.3
7a	1600, 1640 (C=N, C=C)	3.2 (dd, 1H, H-a); 3.8 (dd, 1H, H-b); 5.8 (dd,1H, H-c); 6.8-7.8 (m, 14H, ArH's).	Jab = 16 Jbc = 11 Jac = 8
7b	1600, 1610 (C=N δ C=C)	2.4 (s,3H, CH_3); 3.3 (dd, 1H, H-a); 3.7 (dd, 1H, H-b); 5.8 (dd,1H, H-c); 7.1-8.8 (m, 13H, ArH's).	Jab = 17 Jbc = 11 Jac = 8
7c	1580, 1610 (C=N, C=C)	3.2 (dd, 1H, H-a); 3.8 (dd, 1H, H-b); 5.8 (dd,1H, H-c); 7.2-8.9 (m, 13H, ArH's).	Jab = 17 Jbc = 11 Jac = 8
7d	1580, 1610 (C=N, C=C)	3.2 (dd, 1H, H-a); 3.7 (dd, 1H, H-b); 5.9 (dd, 1H, H-c); 7.2-8.8 (m, 13H, ArH's)	Jab = 18 Jbc = 11 Jac = 8
7e	1600, 1615 (C=N, C=C)	3.3 (dd, 1H, H-a); 4.0 (dd, 1H, H-b); 5.9 (dd, 1H, H-c); 7.2-8.9 (m, 18H, ArH's).	Jab = 18 Jbc = 11 Jac = 8
8a	1610, 1590 (C=N δ C=C)	2.4 (s, 3H, CH_3); 7.2-8.8 (m, 14H, ArH's)	

Table 3. (Contd.)

Compd. No.	IR υ (cm^{-1})	^{1}H-NMR (δ ppm in CD Cl_3)	J = H_z
8b	1600, 1590 (C=N δ C=C)	7.2-8.8 (m, 14H, ArH's).	
8c	1600, 1590 (C=N δ C=C)	7.0-8.8 (m, 19H, ArH's).	
9a	1610, 1540 (C=N δ C=C); 830 (C-S); 920 (S-N)	3.2 (dd, 1H, H-a); 3.6 (dd, 1H, H-b); 5.7 (dd, 1H, H-c); 6.9-7.9 (m, 14H, ArH's).	Jab = 17 Jbc = 10 Jac = 8
9b	1610, 1540 (C=N δ C=C); 820 (C-S); 915 (S-N)	2.4 (s, 3H, CH_3); 3.3 (dd, 1H, H-a); 3.7 (dd, 1H, H-b); 5.8 (dd, 1H, H-c); 7.1-8.8 (m, 13H, ArH's).	Jab = 17 Jbc = 11 Jac = 8
9c	1600, 1540 (C=N δ C=C); 820 (C-S); 915 (S-N)	3.2 (dd, 1H, H-a); 3.6 (t, 3H, H-b); 5.6 (dd, 1H, H-c); 6.8-8.8 (m, 13H, ArH's).	Jab = 17 Jbc = 10 Jac = 8
9d	1615, 1560 (C=N δ C=C); 830 (C-S); 920 (S-N)	3.2 (dd, 1H, H-a); 3.7 (dd, 1H, H-b); 5.9 (dd, 1H, H-c); 7.2-8.8(m, 13H, ArH's).	Jab = 17 Jbc = 11 Jac = 8

The antimicrobial activity

Antimicrobial activity of the compounds was tested on the following organisms:

1. *Strep. pyog.*
2. *Pseudomonas aerugionasa.*
3. *Bacillus subtilis.*
4. *Escherichia coli.*
5. *Proteus mirabilis.*
6. *Salmonella.*
7. *Saccharomyces cerevisa.*
8. *Candida albican.*

The biological activity was determined according to the cup-plate method adopted with some modifications. Whatman No. 2 filter paper disk (6.5 cm) were impregnated with 200 mg of the compound. The disk was placed on the surface of the cold solid medium petridishes, inoculated with the considered organisms, and then incubated at 5^{o}C for one hr, to allow good diffusion and then transferred to an incubator at 28^{o}C and left for 24 hr.

The sensitivity of microorganisms to the compound is identified in the following manner:

+++ = highly sensitive (inhibition zone 1.2 – 1.5 mm)

++ = fairly sensitive (inhibition zone 1.2 – 0.9 mm)

\+ = slightly sensitive (inhibition zone 0.9 – 0.6 mm)

\- = not sensitive

A summary of the biological activity results is shown in Table 4.

Table 4. Antimicrobial activity of selected compounds

Test organism	Compounds					
	3b	**4c**	**5b**	**6a**	**8b**	**9c**
1	++	-	+	+	+	+
2	+++	-	-	+	+	+
3	++	+	+	-	+	+
4	++	-	+	-	-	+
5	+	-	+++	-	++	+
6	++	-	+	-	++	-
7	+	-	-	-	-	-
8	++	+	-	+	-	+++
9	++	+	-	-	-	+

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تحضير ودراسة التأثيرات الحيوية للعديد من مشتقات ٣- أريل -٥(٩-فينانثريل) -أزولات الجديدة

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ملخص البحث. يتفاعل فينانثريل شالكون (١) مع هيدرات الهيدرازين وفينيل هيدرازين ليعطي مشتقات ٢ـ بيرازولين (٣) و(٤) على التوالي. أسيلة (٣) تعطي المركب (٥) الذي يمكن تحضيره أيضا من المركب (١). أكسدة المركب (١) تعطي مشتقات الأوكسيران (٦). كذلك تم تحضير مشتقات الأيزوإكسازولين (٧)والذي بدوره يتفاعل مع ن ـ برومو سكسينميد ليعطي مشتقات الإكسازول (٨) وهذه المشتقات تم تشييدها أيضا من الشالكونات (١) ومشتقات الأكسيران (٦). ويتفاعل المركب (٧) مع خامس كبريتيد الفوسفور ليعطي مشتقات ٢ـ أيزوثيازولين (٩) . وتمّت دراسة التأثيرات الحيوية لبعض هذه المركبات المشيّدة .