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Synthesis and Antibacterial Activity of Oximes, Semicarbazones and Phenylhydrazones

MAJED M. HANIA
Chemistry Department, The Islamic University, Gaza, P.O. Box 108, Gaza, Palestine

A series of new oximes, semicarbazones and phenyl hydrazones have been synthesized from *o*-chloroacetophenone, *p*-methylacetophenone, and *p*-methoxybenzaldehyde and their antibacterial activity has been studied against *E. coli* which gave different results of activity.

Key Words: Synthesis, Antibacterial activity, Oximes, Semicarbazones, Phenylhydrazones.

INTRODUCTION

Oxime, semicarbazone, phenylhydrazone, thiosemicarbazone, hydrazone derivatives are reported to be active as antibacterial[1–5], antitubercular[6] and antilepral[7] compounds. In continuation of our work on the synthesis of oximes, semicarbazones and phenylhydrazones[1], which have been synthesized from *o*-chloroacetophenone, *p*-methylacetophenone and *p*-methoxybenzaldehyde the methods for the synthesis of oximes, semicarbazones and phenylhydrazones have been reported respectivly[3–5] and characterized by IR data and then tested for their antibacterial activity against *E. coli* bacteria. This testing has been done by the methods described by Broth[8] using different concentrations.

EXPERIMENTAL

Melting points were determined in open capillaries and are uncorrected. IR spectra were recorded in KBr on Perkin-Elmer 883 spectrometer. *o*-Chloroacetophenone, *p*-methylacetophenone and *p*-methoxybenzaldehyde were obtained from sigma-Aldrich Ltd. and used without further purifcation.

All compounds were tested for their antibacterial activity against negative bacteria *E. coli* at different concentrations using Broth dilution susceptibility test[8].

Preparation of *o*-chloroacetophenone oxime (1), *p*-methylacetophenone oxime (2) and *p*-methoxybenzaldehyde oxime (3)

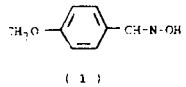
Ketone or aldelyde (0.02 mol) was dissolved in 15 mL ethanol and was added to aqueous solution of hydroxylamine hydrochloride (0.08 mol) and sodium acetate (0.1 mol); the mixture was heated at 80–90°C for 4 h and then left to cool. The precipitate was collected and purified by crystallization from ethanol to give 56, 14 and 27% yield respectively.

Preparation of *o*-chloroacetophenone semicarbazone (4), *p*-methylacetophenone semicarbazone (5) and *p*-methoxybenzaldehyde semicarbazone (6)

Ketone or aldehyde (0.002 mol) was dissolved in 15 mL ethanol and was added to aqueous solution of semicarbazide hydrochloride (0.01 mol); the mixture was heated at 80–90°C for 4 h and then left to cool. The precipitate was collected and purified by crystallization from ethanol to give 45, 79 and 48% yield respectively.

Preparation of *o*-chloroacetophenone phenylhydrazone (7), *p*-methylacetophenone phenylhydrazone (8) and *p*-methoxybenzaldehyde phenylhydrazone (9)

Ketone or aldehyde (0.02 mol) was dissolved in 15 mL ethanol and was added to aqueous solution of phenylhydrazine (0.03 mol); the mixture was heated at 80–90°C for 4 h and then left to cool. The precipitate was collected and purified by crystallization from ethanol to give 27, 56 and 37% yield respectively.



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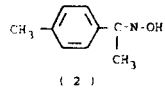

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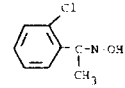

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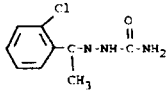

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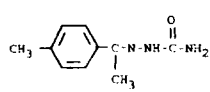

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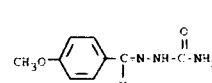

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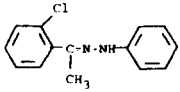

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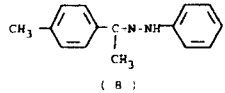

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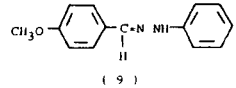


(9)

RESULTS AND DISCUSSION

Oximes (**1, 2** and **3**), semicarbazones (**4, 5** and **6**) and phenylhydrazones (**7, 8** and **9**) were prepared from *o*-chloroacetophenone, *p*-methylacetophenone and *p*-methoxybenzaldehyde which gave a good crystalline yield. The analytical and infrared spectral data are presented in Table-1.

Our antibacterial activity results have been studied to show absolutely different results between oximes, semicarbazones and phenylhydrazone. Oximes showed good activity against –ve bacteria *E. coli*, but semicarbazone and phenylhydrazone showed poor activity against –ve bacteria (Table-2); this might come from different substituents and the functional groups on benzene ring.

Comparison of oximes antibacterial activity against –ve *E. coli* with previous work[1] to show how 2-acetylpyrrole and 2-formylpyrrole oximes did not show antibacterial activity. This might come from the delocalization of the unshared electrons of the pyrrole ring (**Scheme-1**), which produce a negative charge on the

nitrogen of the oxime which might couse repaltion with the body of –ve *E. coli* bacteria which might prevent the inhibition (Table-2).

TABLE-1
ANALYTICAL AND SPECTRAL DATA OF COMPOUNDS

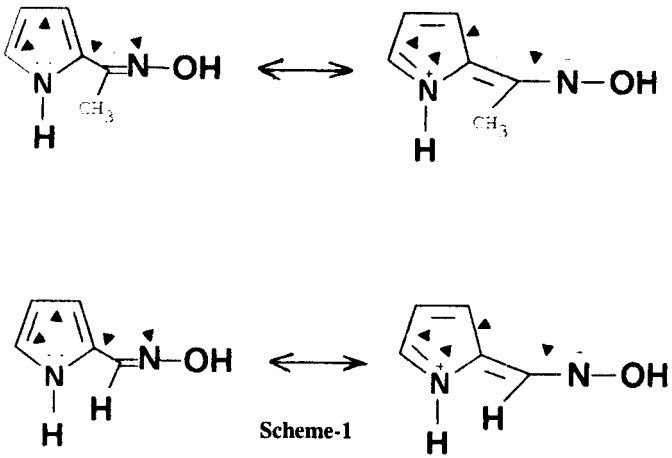
Compound No.	m.p. (°C)	m.f.	IR band (cm^{-1})
1	44–46	$C_8H_9NO_2$	1688 ν(C=N), 3412 ν(O—H), 1118 ν(N—O)
2	84–85	$C_9H_{11}NO$	1651 ν(C=N), 3282 ν(O—H), 1120 ν(N—O)
3	107–108	C_8H_8NOCl	1634 ν(C=N), 3309 ν(O—H), 1134 ν(N—O)
4	184–185	$C_9H_{10}N_3OCl$	1582 ν(C=N), 3215 ν(N—H), 1734 ν(C=O)
5	208–209	$C_{10}H_{13}N_3O$	1582 ν(C=N), 3215 ν(N—H), 1734 ν(C=O)
6	181–182	$C_9H_{11}N_3O_2$	1632 ν(C=N), 3280 ν(N—H), 1684 ν(C=O)
7	135–136	$C_{14}H_{13}N_2Cl$	1646 ν(C=N), 3311 ν(N—H)
8	79–80	$C_{15}H_{16}N_2$	1650 ν(C=N), 3300 ν(N—H)
9	121–122	$C_{14}H_{14}N_2O$	1599 ν(C=N), 3313 ν(N—H)

TABLE-2
ANTIBACTERIAL ACTIVITIES OF COMPOUNDS 1–9

Compound No.	200	100	50	25	12.5
1	+ve	+ve	–ve	–ve	–ve
2	+ve	+ve	+ve	–ve	–ve
3	+ve	+ve	+ve	–ve	–ve
4	–ve	–ve	–ve	–ve	–ve
5	–ve	–ve	–ve	–ve	–ve
6	–ve	–ve	–ve	–ve	–ve
7	–ve	–ve	–ve	–ve	–ve
8	–ve	–ve	–ve	–ve	–ve
9	–ve	–ve	–ve	–ve	–ve

COMPARISON OF OXIME DERIVATIVES

Compound names	Antibacterial activity
2-Acetylpyrrole oxime	Poor
2-Formylpyrrole oxime	Poor
o-Chloroacetophenone oxime	Good
p-Methylacetophenone oxime	Good
p-Methoxybenzaldehyde oxime	Good



Scheme-1

Conclusion

Different oximes, semicarbazones and phenylhydrazones from *o*-chloroacetophenone, *p*-methylacetophenone and *p*-methoxybenzaldehyde compounds have been synthesized which also show good antibacterial activity against –ve *E. coli* in case of oxime derivatives, but poor antibacterial activity against –ve *E. coli* in case of semicarbazone and phenylhydrazone derivatives. This might come from the difference in the electron density on the oxime, semicarbazone and hydrazone derivatives, which reflect the influence of mesomeric and inductive effects on the inhibition of *E. coli* bacteria.

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