



Trade Science Inc.

Organic CHEMISTRY

An Indian Journal
Short Communication

OCAIJ, 6(1), 2010 [119-121]

Synthesis, characterization and antimicrobial activities of 5-(substituted phenyl)-{3-[4-(2-phenyl-4-benzylidene-5-oxo-imidazol-1-yl)]phenyl}-4,5-dihydropyrazol

R.A.Shah¹, P.S.Patel*¹, D.K.Trivedi¹, P.J.Vyas²¹Department of Chemistry, Sheth L.H.Science College, Mansa-382845, (INDIA)²Department of Chemistry, Sheth M.N.Science College, Patan-384265, (INDIA)

E-mail : rajiv_mansa@yahoo.co.in; pspatel_mansa@yahoo.co.in; vyaspiyushj@yahoo.com

Received: 9th January, 2010 ; Accepted: 19th January, 2010

ABSTRACT

5-(substituted phenyl)-{3-[4-(2-phenyl-4-benzylidene-5-oxo-imidazol-1-yl)]phenyl}-4,5-dihydropyrazol have been prepared by the refluxation for three hours of 4-benzylidene-1-[4-[3-(substitutedphenyl)prop-2-enoyl]phenyl]-2-phenyl-imidazol-5-one with hydrazine hydrate in presence of ethanol the intermediate 4-benzylidene-1-[4-[3-(substitutedphenyl)prop-2-enoyl]phenyl]-2-phenyl-imidazol-5-one synthesized by the condensation of 1-(4-acetylphenyl)-4-benzylidene-2-phenyl-imidazol-5-one with various aldehydes. © 2010 Trade Science Inc. - INDIA

KEYWORDS

Synthesis;
Hydrazine hydrate;
Oxazolone;
Substitutedphenyl;
Pyrazol.

INTRODUCTION

During the last few decades, a considerable attention has been devoted to synthesis of heterocyclic Compounds and their derivatives possessing such comprehensive bioactivities as antimicrobial^[1-3], anti-inflammatory^[4], analgesic^[5], antitumoral^[6], antihypertensives^[7], anti convulsant^[8] and antiviral^[9] activities.

From the literature, we found that several Pyrazolines and Imidazolones are known to display antimicrobial and therapeutic activities. Literature survey reveals scant mention of the above compounds with antimicrobial properties and hence more and more derivatives are worth tested for the possible medicinal applications. So we have decided to synthesize 5-(substituted phenyl)-{3-[4-(2-phenyl-4-benzylidene-5-oxo-imidazol-1-yl)]phenyl}- 4, 5-dihydropyrazol.

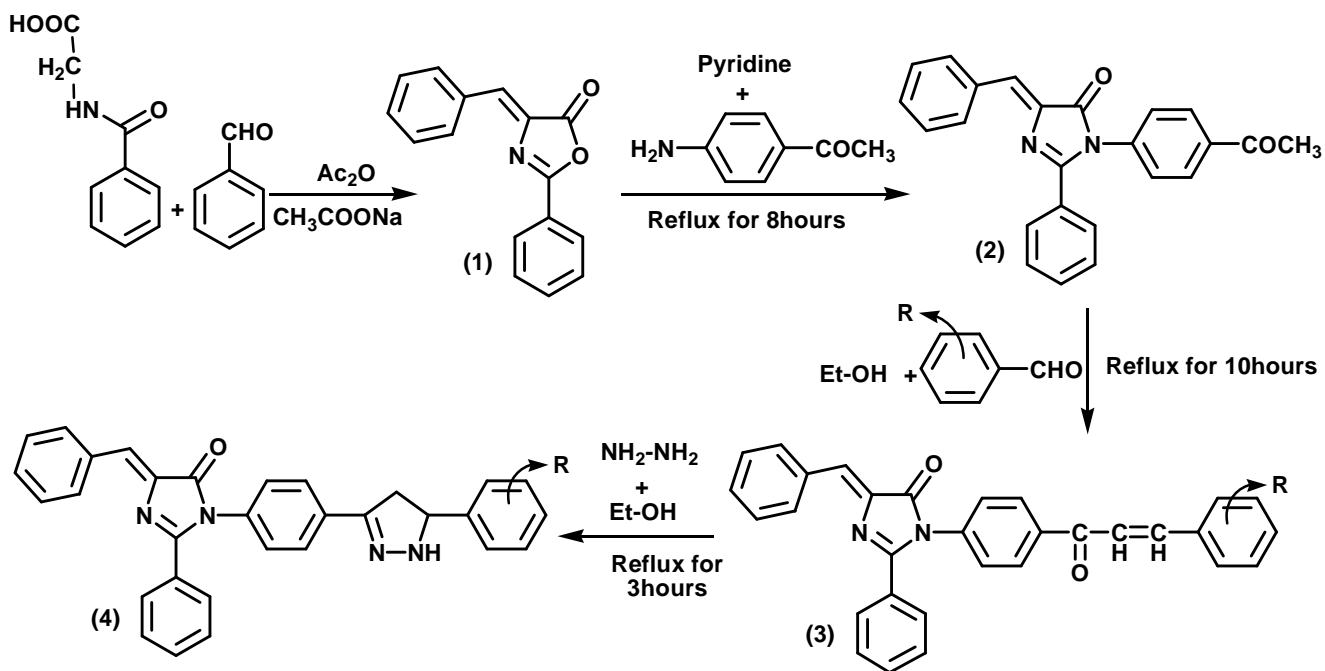
EXPERIMENTAL

Melting points were taken in open capillary tube and were uncorrected. IR spectra (KBr) were recorded on I.R. Spectrophotometer of Buck scientific Model No. 500 and instrument used for NMR Spectroscopy was DUL 13C-1, 300 MHz and tetramethyl silane used as internal standard. Solvent used were CDCl₃ and DMSO. Purity of the compounds were checked by tlc on silica- G plates. Anti microbial activities were tested by Cup-Borer method.

Preparation of 4-benzylidene-2-phenyl-1,3-oxazol-5-one (1)

In a 500ml conical flask equipped with a reflux condenser a mixture of benzaldehyde (27g, 0.25M), hippuric acid (45g, 0.25M), acetic anhydride (77g, 0.75M) and anhydrous sodium acetate (20.5g, 0.25M)

Short Communication



Scheme

TABLE 1 : Physical constant of 5-(substituted phenyl)-{3-[4-(2-phenyl-4-benzylidene-5-oxo-imidazol-1-yl)]phenyl}-4,5-dihydro pyrazol

No.	Sub. No.	R	Molecular formula	Mol.wt. (g/m)	Yield(%)	M.P.°C	Carbon(%)		Hydrogen(%)		Nitrogen(%)	
							Found	required	Found	required	Found	required
1	4a	-4-Cl	C ₃₁ H ₂₃ ClN ₄ O	502.993	56	162	74.00	74.02	4.58	4.61	11.12	11.14
2	4b	-2-Cl	C ₃₁ H ₂₃ ClN ₄ O	502.993	71	188	74.01	74.02	4.57	4.61	11.11	11.14
3	4c	-3-OCH ₃ , -4-OCH ₃	C ₃₃ H ₂₈ N ₄ O ₃	528.600	64	179	74.96	74.98	5.30	5.34	10.58	10.60
4	4d	-2-NO ₂	C ₃₁ H ₂₃ N ₅ O ₃	513.546	67	185	72.46	72.50	4.48	4.51	13.61	13.64
5	4e	-2-OH	C ₃₁ H ₂₄ N ₄ O ₂	484.547	53	132	76.81	76.84	4.96	4.99	11.53	11.56
6	4f	-3-OCH ₃ , -4-OH	C ₃₂ H ₂₆ N ₄ O ₃	514.573	69	198	74.66	74.69	5.06	5.09	10.86	10.89
7	4g	-4-OH	C ₃₁ H ₂₄ N ₄ O ₂	484.547	73	175	76.81	76.84	4.95	4.99	11.53	11.56
8	4h	-4-N(CH ₃) ₂	C ₃₃ H ₂₉ N ₅ O	511.616	55	170	77.45	77.47	5.70	5.71	13.65	13.69
9	4i	-4-OCH ₃	C ₃₂ H ₂₆ N ₄ O ₂	498.574	67	163	77.06	77.09	5.23	5.26	11.21	11.24
10	4j	-3-OCH ₃ , -4-OCH ₃ , -5-OCH ₃	C ₃₄ H ₃₀ N ₄ O ₄	558.626	70	179	73.08	73.10	5.40	5.41	10.01	10.03

was placed and heated on an electric hot plate with constant shaking. As soon as the mixture has liquefied completely, transfer the flask to a water bath and heat for 2 hours. Then add 100 ml of ethanol slowly to the contents of the flask, allow the mixture to stand overnight, filter the crystalline product with solution, wash with 25ml of ice-cold alcohol and then finally wash with 25ml of boiling water, dry at 100°C. The yield of almost pure oxazolone was 64%, m.p. 165°C. Found: C(77.08%) H(4.42%) N(5.60%), Calcd. for C₁₆H₁₁NO₂: C(77.10%) H(4.45%) N(5.62%).

Preparation of 1-(4-acetylphenyl)-4-benzylidene-2-phenyl-imidazol-5-one(2)

In a 250ml conical flask equipped with a reflux condenser a mixture of 4-benzylidene-2-phenyl-1,3-oxazol-5-one(24.92g, 0.1M), 1-(4-aminophenyl)ethanone (13.51g, 0.1M), 25ml pyridine and about one pellet of KOH was placed and was heated on sand bath for 7-8 hours. Then the mixture was poured in ice. The precipitates were collected, washed with 10% HCl and re-crystallized from ethanol. The yield of the product was 75 % and the product melts at 138°C. Found: C(78.65%) H(4.92%) N(7.62%), Calcd. for

Short Communication

TABLE 2 : Antimicrobial activities of 5-(substituted phenyl)-{3-[4-(2-phenyl-4-benzylidene-5-oxo-imidazol-1-yl)]phenyl}-4,5-dihydro pyrazol

Sr. No.	Comp.No.	R	Zone of inhibitions in mm		
			<i>E.coli</i>	<i>S.aureus</i>	<i>C.albicans</i>
1	4a	-4-Cl	17	16	21
2	4	-2-Cl	16	15	17
3	4c	-3-OCH ₃ , -4-OCH ₃	15	18	19
4	4d	-2-NO ₂	14	14	14
5	4e	-2-OH	18	NA	16
6	4f	-3-OCH ₃ , -4-OH	19	17	13
7	4g	-4-OH	20	15	14
8	4h	-4-N(CH ₃) ₂	NA	13	18
9	4i	-4-OCH ₃	15	12	NA
10	4j	-3-OCH ₃ , -4-OCH ₃ , -5-OCH ₃	21	18	20
11	Penicillin	-	18	20	-
12	Kanamycine	-	19	24	-
13	Baycor 25 w.p.	-	-	-	24
14	Amphotericine	-	-	-	21

C₂₄H₁₈N₂O₂: C(78.67%) H(4.95%) N(7.65%); IR (KBr); (cm⁻¹): 3080(= CH-), 3050(-CH Stretch), 1720(>C=Oimidazolone), 1650 (>C=N-), 1605(>C=C<), 1250(C-N).

Preparation of 4-benzylidene-1-{4-[3-(substituted phenyl)prop-2-enoyl]phenyl}-2-phenyl-imidazol-5-one (3)

The solution of 1-(4-acetylphenyl)-4-benzylidene-2-phenyl-imidazol-5-one(3.66g, 0.01M) in absolute ethanol (50ml), substituted benzaldehyde (0.01M) and 2% NaOH (10ml) were added and refluxed for 10 hours. After refluxing the reaction mixture was concentrated, cooled, filtered and neutralized with dil. HCl. The solid residue thus obtained was recrystallized with suitable solvent.

IR (KBr); (3g): (cm⁻¹): 3400(-OH), 3100(= CH-), 2950(-CH Stretch), 1720(>C = O imidazolone), 1650(>C = N-), 1600(>C = C<), 1200 (C-N). NMR; 3f: 3.490, singlate (3H)(-OCH₃), 5.631, singlate (1H) (= CH-vinylic), 6.660-7.902, multiplate (19H) (Ar-H, -CH = CH-) 8.262, singlate(1H) (-OH).

Preparation of 5-(substituted phenyl)-{3-[4-(2-phenyl-4-benzylidene-5-oxo-imidazol-1-yl)]phenyl}-4,5-dihydro pyrazol(4)

A mixture of 4-benzylidene-1-{4-[3-(substituted phenyl)prop-2-enoyl] phenyl}-2-methyl-imidazol-5-one(0.01M) and 99% hydrazine hydrate (0.015M) in ethanol (50ml) was refluxed gently for 3 hours. Then the mixture was concentrated and allowed to cool. The resulting solid was filtered, washed with ethanol and recrystallised from ethanol to give a pale brown solid.

IR (KBr); (4i): (cm⁻¹):3360 (>N-H), 3090 (= CH-), 2910 (-CH), 1720 (>C = O imidazolone), 1620 (>C = N-), 1500(>C = C<), 1460 (>CH₂ pyra zoline), 1250 (N-N), 1270 (C-N), 1160 (-C-O). NMR; (4c): 0.900, dublate (2H)(>CH₂), 1.216, triplate (1H) (>CH-), 3.383, singlate (6H)(-OCH₃), 3.802, singlate (1H)(-NH-), 5.515, singlate (1H) (= CH-vinylic), 7.191-7.240, multiplate (17H) (Ar-H).

ACKNOWLEDGEMENTS

The authors are thankful to the Sheth L.H.Science College, Mansa for providing research facilities. Both the authors Rajiv A.Shah and Pankaj S.Patel are thankful to UGC. Ganeshkhind, pune. for Teacher Research Fellowship.

REFERENCES

- [1] B.S.Holla, R.Gonsalves, S.Shenoy; *Farmaco.*, **53**, 574 (1998).
- [2] S.Ersan, S.Nacak, R.Berkem; *Farmaco.*, **53**, 773 (1998).
- [3] N.N.Gulerman, H.N.Dogan, S.Rollas, C.Johansson, C.C.Elik; *Farmaco.*, **56**, 953 (2001).
- [4] J.R.Maxwell, D.A.Wasdahl, A.C.Wolfson; *J.Med. Chem.*, **27**(12), 1565 (1994).
- [5] G.Turan-Zitouni, Z.A.Kaplancikli, K.Erol, F.S.Kilic; *Farmaco.*, **54**, 218 (1999).
- [6] N.Demirbas, U.A.Demirbas; *Bioorg.Med.Chem.*, **10**, 3717 (2002).
- [7] K.Paulvannan, T.Chen, R.Hale; *Tetrahedron*, **56**, 8071 (2000).
- [8] R.S.Saksena, R.Yasmeen; *Indian J.Chem.B*, **26**, 290 (1987).
- [9] M.I.Husain, M.Amir, E.Singh; *Indian J.Chem.B*, **26**, 251 (1987).