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Reaction 2-R 5-oxo 5-H 6-Ethylcarboxylate 7-phenyl-1,3,4-thiadiazolo- [3,2-a] pyrimidine with Amine

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ABSTRACT

In this article presents Synthesis of 2-R5-oxo 5-H 6-Carboxamid 7-phenyl 1,3,4-thiadiazolo-[3,2-a] pyrimidine through reaction of 2- R 5-Oxo 5-H 6- EthylCarboxilate 7- phenyl-1, 3,4-Thiadiazolo-[3,2-a] pyrimidine with amine. The structures of the compounds obtained are set NMR, ^{13}C, IR- spectroscopy.

Key words: NMR, IR- spectroscopy,^{13}C ,pyrimidine and Thiadiazol.

INTRODUCTION

Condensed derivatives of 1,3,4-thiadiazolo[3,2- a]pyrimidinewere reported to possess a broadspectrum of biological activity[1-4], including antibacterial,antitumor, fungicidal, and herbicidal properties. However,thiadiazoles and their condensed analogs are still in sufficiently studied. In continuation of the search for substances possessing increased ability to permeate through biologicalmembranes of various infectious species [5 – 7] and, in particular, for the new antibacterial drugs in these homologousseries of compounds.

The introduction of a substituent at position 6 of the1,3,4-thiadiazolo [3,2-a] pyrimidine system efficientlyenhances the physiological activity of the oleculef[8-10].This replacement occurs in the reactions of 1,3,4 -thiadiazolo [3,2-a] pyrimidine derivatives withelectrophiles[11-15]. In the present work, we studied the possibilities of thesynthesis of various derivatives of 1,3,4-thiadiazolo [3,2 -a] pyrimidine.

In First we hav synthesized 2-R5-oxo5-H6-Ethyl Carboxilate7-phenyl [1,3,4] thiadiazolo [3,2,-thiadiazole(1)andethyl 2- formyl 3- okco 3- phenyl propanoate(2). (Figure 1)

And more2-R5-oxo5-H6-Ethyl Carboxilate 7-phenyl [1,3,4]thiadiazolo[3,2,-a]pyrimidinereacted with amin(4) until produced 2-R 5-oxo 5-H 6 -Carboxamid 7-phenyl 1,3,4-thiadiazolo-[3,2-a] pyrimidine(5).(Figure 2)

In this regard synthes of 2-R 5-oxo 5-H 6-Carboxamid 7-phenyl -1,3,4-thiadiazolo [3,2-a] pyrimidinedo with The aim of 2-R 5-oxo 5-H 6-ethylcarboxylate 7-phenyl 1 ,3,4-thiadiazolo [3,2-a] pyrimidine and amin in present solvent C_2H_5OH.

RESULT AND DISCUSSION

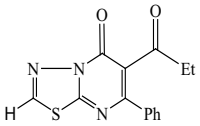
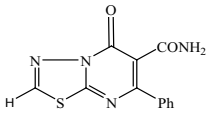
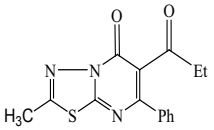
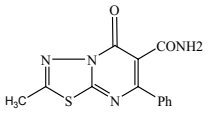
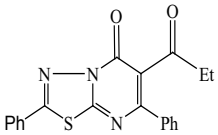
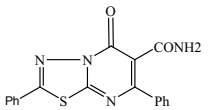
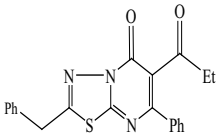
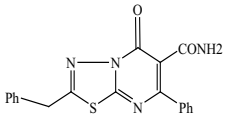
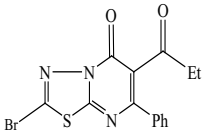
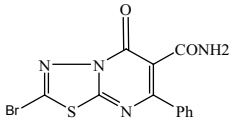
First, we tried synthesisof 2- R 5-oxo 5-H 6-Carboxamid 7-phenyl -1,3,4-thiadiazolo [3,2-a] pyrimidinewith 2-R 5-oxo 5-H 6-ethylcarboxylate 7-phenyl 1 ,3,4-thiadiazolo [3,2-a] pyrimidine and amin in variousAlcohol. The reaction performed in the presence ofAlcohol shown in Table 1, this reaction doesnot take a in the present of catalyst .To show the generality and applicability of this procedure, we treated a wide variety of 2- R 5-oxo

Table 1: Optimization of the reaction conditions

Entry	Solvent	Time(h)	Yield[a](%)
1	H_2O	8	20
2	CH_3OH	4	85
3	C_2H_5OH	4	95
4	C_3H_7OH	5	60
5	C_4H_9OH	6	50

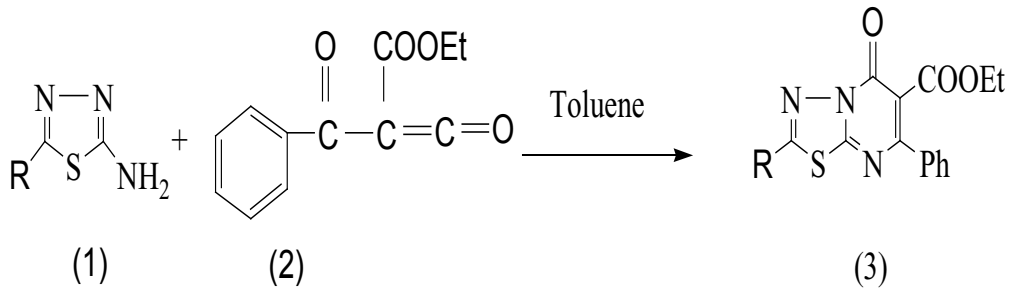
[a]Yields refer to isolated pure products

Table 2: Synthesisof 2- R 5-oxo 5-H 6-Carboxamid 7-phenyl -1,3,4-thiadiazolo [3,2-a] pyrimidine with 2- R 5-oxo 5-H 6-ethylcarboxylate 7-phenyl 1 ,3,4-thiadiazolo [3,2-a] pyrimidine and amina

Entry	Thiadiazol	Amin	Product	Time(h)	Yieldb(%)
1		NH_3		6	82
2		NH_3		8	85
3		NH_3		7	85
4		NH_3		6	90
5		NH_3		5	90

a Reactions were carried out with 2- R 5-oxo 5-H 6-ethylcarboxylate 7-phenyl 1 ,3,4- thiadiazolo [3,2-a] pyrimidine and amin

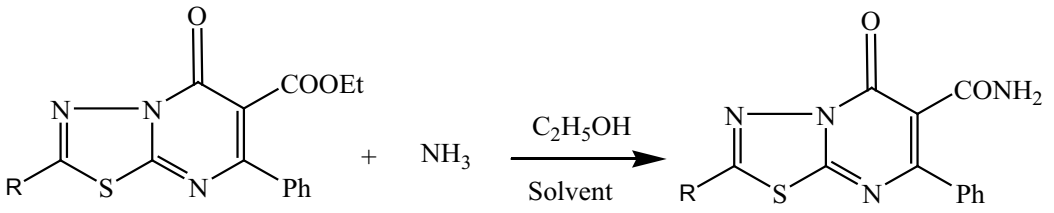
b Yields refer to isolated pure products.



(1) (2) (3)

R:(H, CH_3, Ph- , $PhCH_2$-, Br)

Fig. 1: Synthesis of 2-R7-phenyl 6- ethylcarboxylate 5-oxo 5-H 1,3,4-thiadiazolo [3,2- a]pyrimidine



(3) (4) (5)

R:(H, CH_3, Ph- , $PhCH_2$-, Br)

Fig. 2: Synthesof 2- R 5-oxo 5-H 6-Carboxamid 7-phenyl -1,3,4-thiadiazolo [3,2-a] pyrimidine

5-H 6-ethylcarboxylate 7-phenyl 1 ,3,4- thiadiazolo [3,2-a] pyrimidine and amin in the presence of alcohol ethanol at 78°C and obtained the desirable products in good to excellent yields (Table 2).

EXPERIMENTAL

A mixture of2-R 5-oxo 5-H 6-ethylcarboxylate 7-phenyl 1,3,4- thiadiazolo [3,2-a] pyrimidine (1 mmol), amin (1 mmol) was stirred magnetically at 78°C and the progress of the reaction was monitored by thin-layer chromatography (TLC). The reaction mixture was filtered .In all the cases, the product obtained after the usual work up gave satisfactory spectral data. 2-H 5-oxo 5-H 6-Carboxamid 7-phenyl -1,3,4-thiadiazolo [3,2-a] pyrimidine:^{1}H NMR (400 MHz, $CDCl_3$, δ ppm):6 (s, 2H, NH2) ; 7.14-7.30 (5H, Ph); 7.50 (S, H).^{13}C NMR (100 MHz, $CDCl_3$, δ ppm):118 (C), 126,4 (CH) , 126,4 (CH) ,128(CH), 128.7(CH), 128.7(CH), 136.9(C),140 (C),162,1(C), 163 (C), 168
(C), 172,6.

CONCLUSION

In conclusion, the alcohol has been employed as a mild, and highly efficient solvent system for the convenient preparation of 2- R 5-oxo 5-H 6-Carboxamid 7-phenyl -1,3,4-thiadiazolo [3,2-a] pyrimidine in excellent yields from 2- R 5-oxo 5-H 6-ethylcarboxylate 7-phenyl 1 ,3,4- thiadiazolo [3,2-a] pyrimidine and amin. In addition low cost, recyclable solvent system. The advantages include low cost, mild reaction conditions and reactions carried out at room temperature with excellent yields.

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REFERENCES

1. Japan Patent Application No. 63-10794 (1987); Ref. Zh. Khim.,110402P (1989).
2. US Patent No. 4742063 (1987); Ref. Zh. Khim., 10401P (1989).
3. US Patent No. 4866064 (1989); Ref. Zh. Khim., 70384P (1991).
4. Ì. Suiko, S. Hayashida, and S. Nakatsu, Agric. Biol. Chem., 46(11), 2691 – 2695 (1982).
5. S. S. Sattorov, M. A. Kukaniev, Z. G. Sanginov, et al., Proc. Rep.Conf."Advances in Chemistry and Chemical Technology." [in Russian], Dushanbe pp. 142 – 144 (2002).
6. R. A. Gobum and R. A. Glennon, *J. Med. Chem.,* **17**(9): 1025-1027 (1974).
7. F. Russo, A. Santagati, M. Santagati, *et al., Farmaco. Ed. Sc.*,**42**(6): 437-447 (1987).
8. M.Suiko and K.Maekawa, *Agric.Biol.Chem.,* **41**: 2047 (1977).
9. M.Suiko, E.Taniguchi, K.Maekawa, and M.Eto, *Agric.BioL Chem.*, **43**: 741 (1979).
10. M.Suiko, E.Taniguchi, K.Maekawa, and M.Eto, *BioLChem.,* **43**: 747 (1979).
11. S.Sh.Shukurov, M.A.Kukaniev, I.M.Nasyrov, L.S.Zakharov, and R.A.Kamkhanov, Zh. Obshch. ghim., (63, 2320 [Russ. J. Gen. Chem., 1993, 63(Engl.Transl.)] (1993).
12. A. Akbarzadeh, R. Soleymani, M. Taheri and M.K.C. Ali, *Orient J. Chem.*, **28**(1): 153-164 (2012).
13. R. Moradivalikbori, Y. Hozhiboevy and Z. Heidarnezhad, *Orient J. Chem.*, **29**(4): 1647-1650 (2013).
14. S.Sh.Shukurov, M.A.Kukaniev, M.Nasyrov, L.S.Zakharov, and R.A.Karakhanov, lay. Akad. Nauk, Set.Khim., 1994, 908 [Russ. Chem. BAIL, t994, 43,854 (Engl.Transl.)L.
15. Y.Suichi, M.Tamotsu, A.Shunzo, F.Hiroyuki, M.Hiroo,M.Mikio, R.Reimei, T.Watan ~, and 1.Sumiro, *Chem.and Pharm. Bull.*, **40**: 3391 (1992).