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NOVEL SYNTHESIS OF OXADIAZOLE DERIVATIVES WITH PYRIMIDINE MOIETY

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ABSTRACT: A series of different 6-Methyl - 4- aryl -5 - (5-aryl - 1,3,4-oxadiazole-2-yl) -1,2,3,4-tetrahydro pyrimidin-2- (1H) - one / thione (IIIa-g) have been synthesised from Ethyl - 6- methyl- 2- oxo / thioxo – 4-substituted phenyl-1,2,3,4-tetrahydro pyrimidine-5-carboxylate (Ia-g) followed by reaction with hydrazine hydrate in ethanol gave 6 - methyl-2-oxo / thioxo – 4 -substituted phenyl - 1,2,3,4-tetrahydro pyrimidine-5-carbo hydrazide (IIa-g) by means of Microwave irradiation for 2-4 mins and giving excellent yield in short reaction time, are notable advantages of this method. The structure elucidations of all the synthesised compounds have been accomplished by elemental analysis, IR, NMR and Mass spectroscopic method.

INTRODUCTION: Five membered heterocycle having three heteroatoms like oxadiazole known to have different biological activities such as Antibacterial [1], Anti-inflammatory [2], Anti-convulsant [3], Analgesics [4] activity. These activities are due to the presence of the -N=C-O-linkage and hence oxadiazole and its derivatives have attracted wide attention of chemist for preparation of different biological active drug. There are four possible isomers of oxadiazole depending on the position of nitrogen atom in the ring namely 1, 2, 3-, 1, 2, 4-, 1, 2, 5- and 1, 3, 4-oxadiazoles. Out of these 1, 3, 4-oxadiazoles are found to be most potent biologically [5-12] could become new drug for market in future. Similarly, pyrimidine derivatives are important class of heterocyclic compound due to their therapeutic and pharmacological properties and used as calcium channel blockers and alpha-1α-antogonists.

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The biological and synthetic significance places this scaffold at a prestigious position in medicinal chemistry research so we have developed an operationally simple, inexpensive, efficient and environmental benign protocol for synthesis. In present work, we have developed rapid and operationally simple method for synthesis of different oxadiazoles with pyrimidine nucleus.

MATERIALS AND METHODS: All chemicals were of synthetic grade (S.D. Fine Chem. Ltd. Mumbai, India). Melting point was determined by open capillary method and is uncorrected. Products were recrystalised from ethanol as a solvent. The purity of compound checked by the TLC on silica gel G plates and was purified by column chromatography on silica gel (60-120 mesh). The microwave used for the synthesis is of the LG-Little Chef MS-192 W. The compounds were characterised by using IR, ^{1}H NMR and Mass spectral analysis. The IR spectra were recorded on Perkin-Elmer spectrum in form of KBr pellet .^{1} H NMR was recorded in $CDCl_3$ on Perkin Elmer R-32 spectrum using TMS as internal standard. Mass spectrum was recorded on EI-shimadzu GC-MS spectrometer. All the compounds were analysed for C, H and N on Carlo-Erba elemental analyser.

EXPERIMENTAL SECTION:

Ethyl -6- methyl- 2- oxo/thioxo- 4- substituted phenyl- 1, 2, 3, 4-tetrahydropyrimidine-5-carboxylates:

I(a-g): A mixture of substituted aldehyde (0.01 mol) ethylacetoacetate (0.015 mol),urea /Thiourea (0.01 mol) and concentrated H_2SO_4 (1 – 5 drops) in absolute ethanol (10 ml) were taken in a borosil beaker (250ml) was zapped inside the microwave oven for a period of 3 -4 min (at 160w) the reaction mixture was then allowed to stand at room temperature, and then poured on ice . The product formed was filtered, washed with water, dried and recrystalized from ethanol (**Table 1**).

TABLE 1: PHYSICAL AND ELEMENTAL ANALYSIS OF THE SYNTHESIZED COMPOUNDS (I a-g)

Comp.	R	X	M.P. ^{0}C	Yield %	Mol. Formula	Elemental Analysis Calc. (found)%		
						C	H	N
Ia	-H	O	197	86	$C_{14}H_{16}O_3N_2$	64.61 6.15	(64.60) (6.13)	10.77 (10.76)
Ib	-H	S	170	80	$C_{14}H_{16}O_2N_2S$	60.87 (60.88)	5.80 (5.79)	10.14 (10.13)
Ic	-OH	O	150	82	$C_{14}H_{16}O_4N_2$	60.87 (60.85)	5.80 (5.78)	10.14 (10.15)
Id	-OH	S	120	85	$C_{14}H_{16}O_3N_2S$	57.53 (57.52)	5.48 (5.47)	9.59 (9.55)
Ie	-OCH_3	S	94	87	$C_{15}H_{18}O_3N_2S$	58.82 (58.80)	5.88 (5.80)	9.15 (9.14)
If	-p-Cl	O	202	88	$C_{14}H_{15}O_3N_2Cl$	57.04 (57.18)	5.88 (5.80)	9.15 (9.14)
Ig	-p-Cl	S	70	87	$C_{14}H_{15}O_2N_2ClS$	54.19 (54.18)	4.84 (4.85)	9.03 (9.00)

6-methyl-2-oxo/thioxo-4-substituted phenyl-1, 2, 3, 4-tetrahydropyrimidine-5-carbo hydrazide:

II(a-g): The compound I (0.01 mol) in ethanol and hydrazine hydrate (0.99%, 0.015 mol) were taken in borosil beaker (250 ml) the reaction mixture zapped inside the microwave oven for a period of 2 -3 min. (at 160w). Then reaction mixture allowed to cool for a while after some time mixture was poured on ice. Product formed filtered, washed with water, dried and recrystalized from ethanol (**Table 2**).

TABLE 2: PHYSICAL AND ELEMENTAL ANALYSIS OF THE SYNTHESIZED COMPOUND (IIa-g)

Comp.	R	X	M.P. ^{0}C	Yield %	Mol. Formula	Elemental Analysis Calc. (found) %		
						C	H	N
IIa	-H	O	202	82	$C_{12}H_{14}O_2N_4$	58.54 (58.53)	5.69 (5.700	22.76 (22.75)
IIb	-H	S	190	79	$C_{12}H_{14}ON_4S$	54.96 (54.90)	5.34 (5.31)	21.37 (21.38)
IIc	-OH	O	192	78	$C_{12}H_{14}O_3N_4$	54.96 (54.96)	5.34 (5.30)	21.37 (21.38)
IId	-OH	S	140	85	$C_{12}H_{14}O_2N_4S$	51.80 (51.79)	5.03 (5.00)	20.14 (20.13)
IIe	-OCH_3	S	130	84	$C_{13}H_{16}O_2N_4S$	53.42 (53.40)	5.48 (5.46)	19.96 (19.97)
IIf	-p-Cl	O	214	80	$C_{12}H_{13}O_2N_4Cl$	51.34 (51.30)	4.63 (4.64)	19.96 (19.97)
IIg	-p-Cl	S	150	81	$C_{12}H_{13}ON_4ClS$	48.57 (48.53)	4.38 (4.33)	18.89 (18.91)

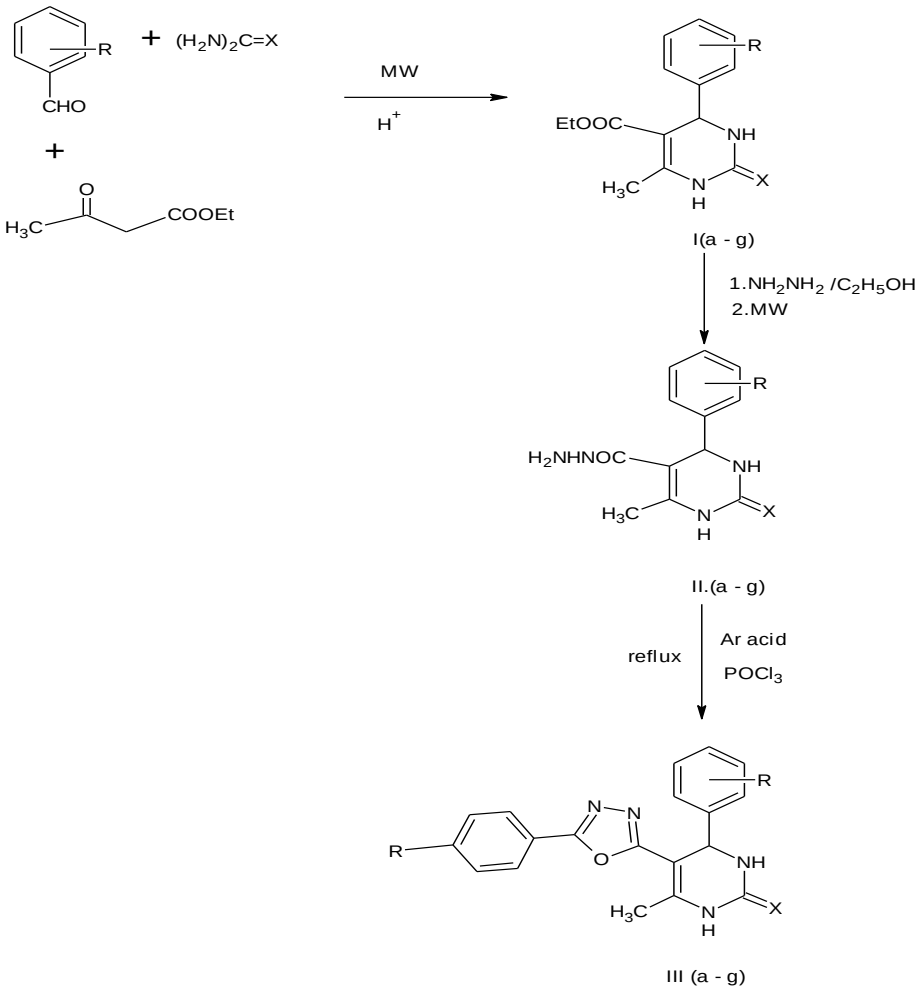
6-methyl-4-aryl-5-(5-phenyl-1, 3, 4-oxadiazole -2-yl)-1, 2, 3, 4-tetrahydropyrimidine-2-(1H)-one / thione:

III(a-g): Carbohydrazides II (0.02 mol) and substituted aromatic acid (0.02 mol) in $POCl_3$ were taken in Round bottom flask. The reaction mixture refluxed for 8- 10 hrs. The reaction mixture was cooled and poured on crushed ice and neutralised with 20% $NaHCO_3$ solution gave solid which was filtered, dried and recrystallized from methanol (**Table 3**).

TABLE 3: PHYSICAL AND ELEMENTAL ANALYSIS OF THE SYNTHESIZED COMPOUND (IIIa-g)

Comp.	R	X	Ar	M.P. ^{0}C	Yield %	Mol. Formula	Elemental Analysis Calc. (found) %		
							C	H	N
IIIa	-H	O	$-C_6H_5$	142	80	$C_{19}H_{16}O_2N_4$	68.67 (68.63)	4.82 (4.80)	16.87 (16.89)
IIIb	-H	S	$-C_6H_5$	90	82	$C_{19}H_{16}ON_4S$	65.51 (65.52)	4.59 (4.50)	16.09 (16.00)
IIIc	-OH	O	$-C_6H_5$	210	85	$C_{19}H_{16}O_3N_4$	65.51 (65.50)	4.59 (4.58)	16.09 (16.08)
IIId	-OH	S	$-C_6H_5$	200	84	$C_{19}H_{16}O_2N_4S$	62.63 62.60)	4.39 (4.38)	15.38 (15.32)
IIIe	$-OCH_3$	S	$-C_6H_5$	90	83	$C_{20}H_{18}O_2N_4S$	63.49 63.48)	4.76 (4.75)	14.81 (14.79)
IIIf	-p-Cl	O	$-C_6H_5$	170	88	$C_{19}H_{15}O_2N_4Cl$	62.21 (62.20)	4.09 (4.01)	15.28 (15.25)
IIIg	-p-Cl	S	$-C_6H_5$	62	87	$C_{19}H_{15}ON_4SCl$	59.60 (59.62)	3.92 (3.90)	14.64 (14.64)

RESULTS AND DISCUSSION: In this present work, synthesis of some new Oxadiazole derivatives with pyrimidine moiety have been reported from corresponding different hydrazide derivatives (IIa-g, Table 2). Initially, substituted pyrimidine carboxylate I(a-g) were prepared by our earlier reported method i.e. Hantsch synthesis which were treated with hydrazine hydrate in ethanol to furnish the corresponding substituted hydrazide derivatives by microwave irradiation (IIa-g) followed by reflux with aromatic acid in $POCl_3$ predicts 6-methyl- 4- aryl- 5- (5-phenyl- 1, 3, 4-oxadiazole -2 -yl)- 1, 2, 3, 4 - tetrahydropyrimidine-2- (1H)-one/thione: IIIa-g (**Table 3, Scheme I**)).



SCHEME 1

The newly synthesized compounds I(a-g), II(a-g) and III(a-g) were established on the basis of IR, ^{1}H NMR and MASS spectroscopic method. The IR spectra of the compounds (IIa-g) showed absorption band at 1664-1672 cm^{-1} indicates presence of amide group and in IIIa-g, absence of absorption band at 1664-1672 cm^{-1} and at 1270cm^{-1} due to presence of C-O-C group and indicating the formation of product. In ^{1}H NMR spectra, a peak observed at 4.53 ppm due to presence of -NH_2 group in IIa-g. While in oxadiazole derivatives, absence of peak at 4.53 due to –NH_2 proved the structure of the products, the mass spectra of the substituted oxadiazole with pyrimidine derivative were showed molecular ion peak corresponding to their molecular formula. The IIIf and IIIg compound shows [M+] and [M^+ +2] peak at m/z 366.5(M^+), 368.5(M^++2) and 382.5(M^+), 384.5(M^++2) showing presence of halogen respectively and peak at 35.5 and 37.5 confirms presence of Chlorine in the ratio 1:3.

TABLE IV: IR, NMR AND MASS SPECTRAL ANALYSIS (Ia-IIIg)

Comp. No.	IR (KBr)	NMR($CDCl_3$)	MASS (m/z)
Ia	Vmax, 3226.33 (-NH), 1718(>C=O, ester), 1668.98(>C=O, amido), 1640 (>C=C<) cm^{-1}.	δ ,1.25 (3H,t,-CH_3), 2.31 (3H,s,-CH_3), 4.2(2H,q, -CH_2), 5.4(1H,s,-CH), 5.9(1H,s,-NH), 7.2-7.4(5H,m ,Ar-H), 8.4 (1H, s, -NHCO) ppm.	-
Ib	Vmax, 3226.30 (-NH), 1728(>C=O), 1641(>C=C<), 1248(>C =S), cm^{-1}.	δ ,1.31(3H,t,-CH_3), 2.38(3H,s,-CH_3), 4.52(2H,q,CH_2), 5.45(1H,s, -CH), 5.83(1H,s,NH), 7-7.5(5H,m,Ar-H), 8.2 (1H,s,NHCO) ppm.	-
Ic	Vmax, 3610 (Ar-OH), 3226.33(-NH), 1710.12 (>C=O, ester), 1673.98(>C=O, amido), 1640(>C=C<), Cm^{-1}.	δ ,1.28(3H,t,-CH_3), 2.39(3H,s,- CH_3), 4.5(2H,q,-CH_2), 5.51(1H,s,-CH), 5.9(1H,s,NH), 7.2-7.5(4H,m,Ar-H), 8.9(1H,s,NH), 9.52(1H,s ,Ar- OH) ppm.	-
Id	Vmax, 3610 (Ar-OH), 3226.33 (-NH), 1730 (>C=O) , 1640 (>C=C<), 1240 (>C=S)Cm^{-1}.	δ ,1.23(3H,t,-CH_3), 2.48 (3H,s,-CH_3), 4.4(2H,q,-CH_2), 5.53(1H,s,CH), 5.6(1H,s,-NH), 7-8(4H,m,Ar-H), 8.5(1H,s,NH), 9.8(1H,s,Ar- OH) ppm.	-
Ie	Vmax, 3230.33(-NH), 1725 (>C=O), 1650 (>C=C<), 1241(>C=S) Cm-^{1}.	δ ,1.4(3H,t,CH_3), 2.50(3H,s,CH_3), 3.5(2H,q,-CH_2), 4.1(3H,s,OCH_3), 5.52(1H,s,CH),5.8(1H,s,NH), 7-8 (4H,m,Ar-H) ,8.23 (1H,s, NHCO) ppm.	-
If	Vmax, 3210.33(-NH), 1728 (>C=O,ester), 1684 (>C=O, amido),1650 (>C=C<), 780(-C-Cl) Cm^{-1}.	δ ,1.4(3H,t,-CH_3), 2.50(3H,s,-CH_3), 3.5(2H,q,-CH_2), 5.52 (1H,s,-CH), 5.8(1H,s,-NH), 7.1-7.4(4H,m,Ar-H). 8.08 (1H,s,-NHCO) ppm.	m/z 294.5(M^+), 296.5(M^++2).
Ig	Vmax, 3225.33(-NH), 1718 (-C=O), 1650 (>C=C<), 1248(>C=S), 782 (-C-Cl), Cm^{-1}.	δ, 1.43(3H,t,-CH_3), 2.50 (3H,s,-CH_3), 4.5(2H,q,CH_2), 5.53(1H,s,CH), 5.8(1H,s,NH), 7.1-7..4(4H,m,Ar-H). 8.2(1H,s,-NHCO) ppm.	m/z 310(M^+), 312(M^++2).
IIa	Vmax ,3213.33(-$NHNH_2$), 3049 (Ar-H), 1664 (amido,>C=O), 1648 (>C=C<), Cm^{-1}.	δ, 2.28(3H,s,-CH_3), 4.2(2H,d,NH_2), 5.50 (1H,s,-CH), 7.1-7.3(5H,m,Ar-H) 7.9(1H,s,NH), 8.4(1H,s,NHCO).ppm.	-
IIb	Vmax , 3220.33(-$NHNH_2$), 3049 (Ar-H), 1670 (amido,>C=O), 1650 (>C=C<), 1244 (>C=S). Cm-^{1}.	δ, 2.3(3H,s,-CH_3), 4.27(2H,d,NH_2), 5.53 (1H,s,-CH), 7.1-7.3(5H,m,Ar-H) 7.5(1H,s,NH), 8.3(1H,s,-NHCO) ppm.	-
IIc	Vmax, 3332 (Ar-OH), 3223 (-$NHNH_2$), 3049 (Ar-H), 1670(amido->C=O), 1654 (>C=C<), Cm-^{1}.	δ, 2.40(3H,s,-CH_3), 4.3(2H,d,-NH_2),5.47(1H,s,Ar-OH), 5.51(1H,s,-CH), 6.6(1H,s,-NH), 7.1-7.3(4H,m,Ar-H). 7.8(1H, s,NH), 8.3(1H,s,-NHCO) ppm.	-
IId	Vmax, 3432 (-OH) ,3231.27 (-$NHNH_2$), 3050(Ar-H), 1672 (amido->C=O), 1653(>C=C<), 1240 (>C=S). Cm-^{1}.	δ, 2.39(3H,s,-CH_3), 4.28(2H,d,-NH_2), 5.5(1H,s,Ar-OH), 5.53(1H,s,-CH), 6.67(1H,s,-NH), 7.1-7.3(4H,m,Ar-H).7.82(1H,s,NH), 8.34(1H,s,-NHCO) ppm.	-
IIe	Vmax, 3332(-$NHNH_2$), 3049(Ar-H), 1668 (amido,>C=O), 1604 (>C=C<), 1242 (>C=S). Cm-^{1}.	δ, 2.34(3H,s,-CH_3),4.3(2H,s,-NH_2), 4.48(3H,s,OCH_3), 5.45(1H,s,-CH), 5.82(1H,s,NH), 7.1-7.3(4H,m,Ar-H), 7.8(1H,s,NH),8.13(1H,s,-NH) ppm.	-
IIf	Vmax, 3330.33(-NH), 3049(Ar-H), 1672(amido-C=O), 1615(>C=C<), 838 (C-Cl) Cm-^{1}.	δ, 2.31(3H,s,-CH_3), 4.62(2H,d,-NH_2), 5.50(1H,s,CH), 5.76(1H,s,-NH), 7.1-7.3(4H,m ,Ar-H). 7.7(1H, s,-NH), 7.9(1H, s,-NHCO) ppm.	m/z 280.5(M^+), 282.5(M^++).
IIg	Vmax, 3430.33(-NH), 3049 (Ar-H), 1648(amido-C=O), 1634 (>C=C<), 1258 (>C=S) 833 (-C-Cl), Cm-^{1}.	δ, 2.35(3H,s,-CH_3), 4.66(2H,d,-NH_2), 5.53(1H,s,-CH), 5.76(1H,s,-NH), 7.1-7.3(4H,m ,Ar-H). 7.8(1H, s,-NH), 8.23(1H,s,-NHCO), ppm .	m/z 296.5(M^+), 298.5(M^++2).

IIIa	Vmax, 3233(-NH), 1627 (>C=C<), 1698.33(>C=O), 1604 (>C=N), 1270(C-O-C), 1062 (N-N), 1070 (- C-O). Cm^{-1}.	δ, 2.48(3H,s,CH_3), 5.5(1H,s,CH), 6.4(1H,s,-NH), 7.2-8.1(10H,m, Ar-H). 8.7(1H,s,NH), ppm.	m/z 332.3.(M^+)
IIIb	Vmax, 3233(-NH), 1604(>C=N), 1527(>C=C<), 1062(N-N), 1070(>C-O),1269 (C-O-C),Cm^{-1}.	δ, 2.28(3H,s,-CH_3), 5.55(1H,s,-CH), 6.33(1H,s,-NH), 7.2-8.1(10 H,m, Ar-H), 8.67(1H,s,NH), ppm.	m/z 347.7(M^+)
IIIc	Vmax, 3312 (>C-OH), 3233 (-NH), 1698.33 (>C=O), 1527 (>C=C<), 1604 (>C=N), 1269 (C-O-C), 1062(N-N), 1070 (-C-O), Cm^{-1}.	δ, 2.48(3H,s,-CH_3), 5.70(1H,s,CH), 5.88(1H,s,-OH), 6.4(1H,s,-NH), 7.1-8.2(9H,m,Ar-H). 8.8(1H,s,-NHCO), ppm.	m/z 348.1(M^+)
IIId	Vmax, 3314 **(**-C-OH), 3213(-NH), 1528(>C=C<), 1600(-C=N), 1270(C-O-C), 1242 (>C=S), 1062(N-N), 1072(C-O) Cm^{-1}.	δ, 2.42(3H,s,-CH_3), 5.69(1H,s,-CH), 5.81(1H,s,-OH), 6.4(1H,s,-NH), 7.1-8.2(9H,m,Ar-H). 8.78(1H, s,-NHCO), ppm.	m/z 356.0(M^+)
IIIe	Vmax, 3233(-NH), 1604(>C=N), 1520(>C=C<), 1269(C-O-C), 1242 (>C=S), 1062 (N-N), 1070(C-O). Cm^{-1}.	δ ,2.45(3H,s, -CH_3), 4.6(3H,s,-OCH_3), 5.65(1H,s,CH), 6.4(1H,s,-NH), 7.1-8.3(9H,m,Ar-H). 8.7(1H,s,-NHCO), ppm.	m/z 378.5(M^+)
IIIf	Vmax, 3233(-NH), 1698.33 (- C=O), 1604 (>C=N), 1062(N-N), 1527(>C=C<), 1352(-C-N), 1041.47(C-O-C), 780 (C-Cl). Cm^{-1}.	δ ,2.48(3H,s,CH_3), 4.61(3H,s,-OCH_3), 5.56(1H,s,CH), 6.3(1H,s,-NH), 7.1-8.2 (9H,m,Ar-H), 8.7(1H,s,-NHCO) ppm.	m/z 366.5(M^+), 368.5(M^++2).
IIIg	Vmax, 3213(-NH), 1698.33 (-C=O), 1604 (-C=N), 1527(>C=C<), 1270 (C-O-C), 1240(- C=S), 1070(C-O), 1062 (N-N), 780(C-Cl). Cm^{-1}.	δ ,2.43(3H,s,CH_3), 4.60(3H,s,-OCH_3), 5.66(1H,s,CH), 6.34(1H,s,-NH), 7.1-8.2 (9H,m,Ar-H), 8.73(1H,s,-NHCO) ppm.	m/z 382.5(M^+), 384.5(M^++2).

CONCLUSION: Keeping in view green approach, we have developed an operationally simple, inexpensive, efficient and environmental benign protocol for synthesis of oxadiazoles with pyrimidine moiety.

Synthesis of pyrimidine carboxylate derivatives were carried out by Hanztsch method followed by hydrazine hydrate in ethanol obtained Carbohydrazides under Microwave irradiation for 2-4 min., followed by cyclisation in $POCl_3$ gave Oxadiazoles.

The merits of the current protocol are:

1. Yields are excellent.
2. Required short reaction time.
3. Easy workup synthesis and operable on large scale.

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