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$(PhIO)_n$ Mediated Annulations of Aromatic Aldehyde *N*-Acylhydrazones for the Synthesis of 1,3,4-Oxadiazoles

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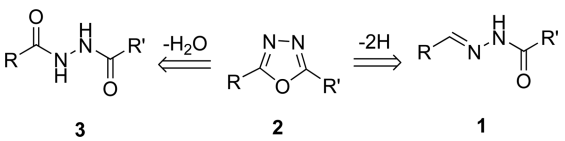
Oxadiazole as a class of five-membered heterocycles has been reported to be biologically versatile compounds due to their metabolic profile. Amongst these heterocycles, 1,3,4-oxadiazole has become an important construction motif for the development of new drugs, furamizole[1] as antibiotics, tiodazosin[2] as antihypertensive agent and raltegravir[3] as anti-retroviral agent have been on market. In addition, 2,5-disubstituted-1,3,4-oxadiazole derivatives have also been used as electron conducting and hole blocking (ECHB) materials for electron deficient and good electron transport properties of oxadiazole rings.[4]

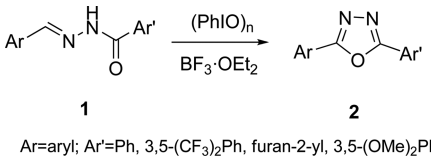
The most common synthetic approach to 1,3,4-oxadiazoles involves cyclodehydration of 1,2-diacylhydrazines 3 (Scheme 1). Typically, this reaction is carried out by using $SOCl_2$,[5] $POCl_3$,[6] as well as others.[7] Although these methods are very useful for the preparation of large quantities of materials due to the ready availability of diacylhydrazines, hydrazides, and hydrazones as starting materials, the reaction conditions tend to be harsh and long reaction times are generally needed.

An alternative route to 1,3,4-oxadiazoles 2 by oxidative cyclization from the corresponding aldehyde *N*-acylhydrazones 1 proceeds (a) with transition metal oxidants such as Pb^{4+}, Mn^{7+}, Fe^{3+}, Ce^{4+} or Cu^{2+} *et al.*[8]; (b) with chloramine T, Trichloroisocyanuric Acid, iodobenzene diacetate (IBD), Dess–Martin reagent (DMP) or other oxidants[9]; (c) electrochemical methods[10] (Scheme 1).

To the best of our knowledge, preparation of 1,3,4-oxadiazoles utilizing iodosobenzene as the oxidizing agent has not been reported yet. In our continuous research program of the oxidation of hypervalent iodine,[11] we were interested in the development of robust and general method to access functionalized 2,5-disubstituted-1,3,4-oxadiazoles.



Scheme 1. General process for the synthesis of 1,3,4-oxadiazoles.



Scheme 2. Oxidative cyclization of *N*-acylhydrazones by $(PhIO)_n$/$BF_3{\cdot}Et_2O$.

It was found that aromatic aldehyde *N*-acylhydrazones 1 reacted with $(PhIO)_n$/$BF_3{\cdot}Et_2O$ at room temperature to efficiently afford 2,5-disubstituted-1,3,4-oxadiazoles 2 in mild to good yields (Scheme 2).

The polymeric character of iodosobenzene makes it less suitable as oxidant than other hypervalent reagents, mainly because of its insolubility in ordinary solvents. Its utility is, however, greatly enhanced when it is used with catalysts, notably boron trifluoride. The electrophilic character of iodine in iodosobenzene is greatly increased by the addition of boron trifluoride,[12] probably because of the *in-situ* formation of the monomeric dipole PhI^{+}-OBF_3^{-}.

N'-Benzylidenefuran-2-carbohydrazide (**1a**) was chosen as the substrate in initial studies (Table 1). When the substrate

Table 1. Optimization of oxidative cyclization

Substrate (mmol)	Solvent (mL)	Oxidant (mmol)	$BF_3{\cdot}Et_2O$ (*ca* 48%) (ml)	Reaction temperature	Reaction time/isolated yield (min/%)
1.0	MeOH (2 mL)	1.0		rt	32/83
1.0	MeOH (2 mL)	1.3		rt	7/80
1.0	$CHCl_3$ (45 mL)	1.3		reflux	60/84
1.0	THF (15 mL)	1.5	1.5	rt	2/89
1.0	THF (6 mL)	1.3	1.5	rt	2/87
1.0	**THF (6 mL)**	**1.3**	**1.0**	**rt**	**3/90**
1.0	THF (6 mL)	1.5	1.0	rt	2/88
1.0	THF (6 mL)	1.3	0.6	rt	25/81
1.0	THF (3 mL)	1.3	1.0	rt	7/86

was mixed with iodosobenzene (1.3 eq) in methanol at room temperature, the reaction was completed in 7 minutes. As Hill and Malacria[13] shown, the initial step is presumably the solvolysis of iodosobenzene affording $PhI(OMe)_2$ which can oxidize *N'*-benzylidenefuran-2-carbohydrazide into 2-(furan-2-yl)-5-phenyl-1,3,4-oxadiazole. When the solvent was changed to $CHCl_3$ at room temperature, $(PhIO)_n$ can't be dissolved and no product appeared. Once the reaction mixture was refluxed for 1 h, the yield of **2a** is 84%, but the ratio of solvent volume and **1a** is 45 mL:1 mmol (**1a**).

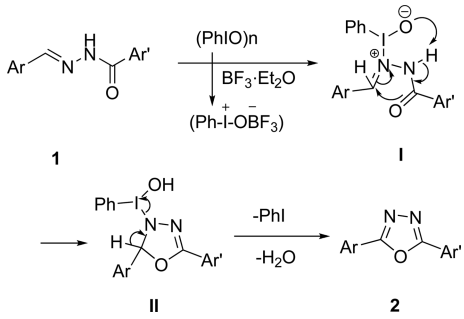
The solvent was changed to THF, iodosobenzene also can't be dissolved. When $BF_3{\cdot}Et_2O$ (*ca.* 48%) was added into the reaction mixture, clear solution appeared immediately and the reaction was completed rapidly at room temperature. As the literature[12] shown, iodosobenzene boron trifluoride which was *in-situ* generated from iodosobenzene and boron trifluoride etherate oxidized acylhydrazones into oxadiazoles. The volume of THF is much less than $CHCl_3$. Reaction time was much longer when $BF_3{\cdot}Et_2O$ solution is less added, which maybe result from the dissolution of iodosobenzene affected by the amount of boron trifluoride. The ratio of substrate and oxidant is 1:1.3 through optimization.

In the reported case, the similar advantage of this reagent was also observed and all of the reported reactions can be finished in 20 minutes at room temperature (Table 2).

A variety of substituents such as NO_2, $(CH_3)_2N$, OMe and CF_3 on the aromatic ring are compatible with this reaction condition. In general, when the aldehyde section contains NO_2 group (Entries **f**, **r**) or $Me_2NC_6H_4$(Entries **g**, **q**), the yields are lower than that of other aldehyde hydrazones with OMe group, possibly due to resonance stabilization by the methoxy group. No product appeared when the aldehyde section just contained OH group (Entry **e**). The substituents of the *N*-acylhydrazones can be substituted benzoyl groups (Entries **h-s**) or heterocycle groups (Entries **a-g**), 2-(furan-2-yl)-5-phenyl-1,3,4-oxadiazole can be prepared from furan-2-carbaldehyde benzohydrazide (Entry **m**) or benzaldehyde furan-2-carboxylic acid hydrazide (Entry **a**) for the symmetry of the 1,3,4-oxadiazole ring. *N*-Furoylhydrazones generally gave higher yields of 1,3,4-oxadiazoles than *N*-substitutedbenzoylhydrazones, the electronic property of the N-substitution plays certain roles in the procedure of cyclization.

Table 2. Oxidative cyclization of aromatic aldehyde *N*-acylhydrazones with $(PhIO)_n/BF_3{\cdot}Et_2O$

Entries	Ar	Ar	Reaction times (min)	Isolated yield (%)
a	Ph	2-Furyl	3	90
b	*o*-ClC_6H_4	2-Furyl	5	93
c	*p*-$MeOC_6H_4$	2-Furyl	4	86
d	2-Furyl	2-Furyl	2	90
e	*p*-OHC_6H_4	2-Furyl		–
f	3-nitroC_6H_4	2-Furyl	12	67
g	*p*-$Me_2NC_6H_4$	2-Furyl	15	38
h	*o*-ClC_6H_4	3,5-$(CF_3)_2$ C_6H_3	12	70
i	2-Furyl	3,5-$(CF_3)_2$ C_6H_3	2	44
j	*p*-$MeOC_6H_4$	3,5-$(CF_3)_2$ C_6H_3	7	72
k	Ph	Ph	3	87
l	*p*-$MeOC_6H_4$	Ph	2	78
m	2-Furyl	Ph	3	58
n	3,5-$MeOC_6H_3$	Ph	12	78
o	*o*-ClC_6H_4	Ph	10	76
p	*p*-$MeOC_6H_4$	3,5-$(OMe)_2$ C_6H_3	5	86
q	*p*-$Me_2NC_6H_4$	3,5-$(OMe)_2$ C_6H_3	7	47
r	3-nitroC_6H_4	3,5-$(OMe)_2$ C_6H_3	8	61
s	2-Furyl	3,5-$(OMe)_2$ C_6H_3	4	84



Scheme 3. The proposed mechanism of the cyclization.

The proposed mechanism is showed following. Polymeric iodosobenzene is depolymerized with BF_3 to generate a higher reactive iodine(III) species,[12] which reacts with the substrate **1** to form an intermediate **I**. After an intramolecular nucleophilic displacement by the nitrogen or oxygen atom, the reaction affords the corresponding 1,3,4-oxadiazole accompanied by the reductive elimination of PhI (Scheme 3).

Experimental Section

Melting points were determined on a X-4 micro-hot stage and are uncorrected. IR spectra were recorded on a FTS 3000 instrument (BIO-RAD). ^{1}H NMR spectra were recorded on Bruker AV 500 MHz instrument using $CDCl_3$ as a solvent, and chemical shifts (d) are in parts per million (ppm) relative to TMS. All solvents were dried.

A round-bottomed flask was charged with aldehyde *N*-acylhydrazone[14] (0.5 mmol), $(PhIO)_n$(0.65 mmol), BF_3 Et_2O (*ca.* 48%, 0.5 mL) and THF (3 mL). This mixture was clear and stirred at room temperature monitored by TLC. When the reaction was complete, The product was purified by flash chromatography (silica gel, ethyl acetate-petroleum ether = 1:4) to afford 2,5-disubstituted-1,3,4-oxadiazoles.

2-(Furan-2-yl)-5-phenyl-1,3,4-oxadiazole (2a, 2m). White solid, mp 99-101 °C (lit.[15] 99-100 °C); IR (KBr): 805, 1021, 1095, 1262, 1521, 1635, 2965 cm^{-1}; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 6.64 (dd, 1H, J_1 = 1.5 Hz, J_2 = 3.5 Hz), 7.26 (t, 1H, J = 8.0 Hz), 7.51-7.56 (m, 3H), 7.67 (d, 1H, J = 1.0 Hz), 8.12-8.14 (m, 2H).

2-(2-Chlorophenyl)-5-(furan-2-yl)-1,3,4-oxadiazole (2b). White solid, mp 93-94 °C; IR (KBr): 799, 1019, 1261 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.63 (s, 1H), 7.25 (d, 1H, J =

2.5 Hz), 7.44 (t, 1H, J = 2.5 Hz), 7.50 (t, 1H, J = 2.5 Hz), 7.58 (d, 1H, J = 7.5 Hz), 7.68 (s, 1H), 8.07 (d, 1H, J = 7.0 Hz).

2-(Furan-2-yl)-5-(4-methoxy-phenyl)-1,3,4-oxadiazole (2c). White solid, mp 129-130 ºC, (lit.[16] 127-129 ºC); IR (KBr): 898, 1103, 1197, 1276, 1319, 1458, 1509, 1627, 2949, 3148 cm^{-1}; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 3.89 (s, 3H), 6.61 (dd, 1H, J_1 = 2.0 Hz, J_2 = 3.5 Hz), 7.02-7.04 (m, 2H), 7.21 (d, 1H, J = 2.5 Hz), 7.66 (d, 1H, J = 1.5 Hz), 8.05-8.08 (m, 2H).

2,5-Di(furan-2-yl)-1,3,4-oxadiazole (2d). White solid, mp 129-130 ºC, (lit.[16] 127-129 ºC); IR (KBr): 752, 804, 895, 1021, 1027, 1076, 1098, 1262, 1449, 1520, 1628, 1645, 2965, 3108, 3125, 3140 cm^{-1}; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 6.63 (dd, 2H, J_1 = 1.5 Hz, J_2 = 3.5 Hz), 7.24-7.26 (m, 2H), 7.67 (s, 2H).

2-(Furan-2-yl)-5-(3-nitrophenyl)-1,3,4-oxadiazole (2f). Yellow solid, mp 176-177 °C. (lit.[17] 178 ºC); IR (KBr): 752, 790, 1011, 1258, 1354, 1620, 1638 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.68 (m, 1H, J = 2.0 Hz), 7.33 (d, 1H, J = 3.5 Hz), 7.72-7.72 (m, 1H), 7.78 (t, 1H, J = 8.0 Hz), 8.41-8.44 (m, 1H), 8.49-8.51 (m, 1H), 8.95 (d, 1H, J = 2.0 Hz).

4-(5-(Furan-2-yl)-1,3,4-oxadiazol-2-yl)-*N*,*N*-dimethylbenzenamine (2g) White solid, mp 162-163 °C; IR (KBr): 805, 1098, 1262, 1506, 1618, 2965, 3106 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 3.07 (s, 6H), 6.60-6.61 (m, 1H), 6.77 (t, 2H, J = 2.0 Hz), 7.18 (q, 1H, J_1 = 0.5 Hz, J_2 = 3.0 Hz), 7.64-7.65 (m, 1H), 7.96-7.98 (m, 2H).

2-(3,5-Bis(trifluoromethyl)phenyl)-5-(2-chlorophenyl)-1,3,4-oxadiazole (2h) White solid, mp 157-159 °C; IR (KBr): 679, 694, 775, 797, 926, 1008, 1110, 1261, 1453 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.46-7.50 (m, 1H), 7.53-7.56 (m, 1H), 7.61-7.63 (m, 1H), 8.07 (s, 1H), 8.12-8.13 (m, 1H), 8.60 (s, 2H).

2-(3,5-Bis(trifluoromethyl)phenyl)-5-(furan-2-yl)-1,3,4-oxadiazole (2i) White solid, mp 127-128 °C; IR (KBr): 695, 710, 774, 846, 901, 1011, 1097, 1140, 1261, 1408, 1517, 1621 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.67-6.68 (m, 1H), 7.34-7.35 (m, 1H), 7.73-7.73 (m, 1H), 8.06 (s, 1H), 8.41-8.44 (m, 1H), 8.58 (s, 1H).

2-(3,5-Bis(trifluoromethyl)phenyl)-5-(4-methoxyphenyl)-1,3,4-oxadiazole (2j) White solid, mp 137-138 °C. (lit.[21] 127-129 ºC); IR (KBr): 698, 711, 795, 837, 960, 1013, 1256, 1275, 1493, 1608 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 3.91 (s, 3H), 7.07 (d, 2H, J = 9.0 Hz), 8.04 (s, 1H), 8.12 (d, 2H, J = 9.0 Hz), 8.57 (s, 2H).

2,5-Diphenyl-1,3,4-oxadiazole (2k) White solid, mp 137-139 °C. (lit.[16] 130-140); IR (KBr): 687, 709, 10695, 1446, 1485, 1549 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.55-7.56 (m, 3H), 8.15-8.17 (m, 2H).

2-(4-Methoxyphenyl)-5-phenyl-1,3,4-oxadiazole (2l) White solid, mp 143-144 °C. (lit.[16] 142 ºC, lit.[17] 146 ºC); IR (KBr): 833, 1017, 1263, 1503, 1616 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 3.89 (s, 3H), 7.04 (dd, 2H, J_1 = 2.0 Hz, J_2 = 7.0 Hz), 7.52-7.54 (m, 3H), 8.09 (dd, 2H, J_1 = 2.0 Hz, J_2 = 7.0 Hz), 8.12-8.14 (m, 2H).

2-(3,5-Dimethoxyphenyl)-5-phenyl-1,3,4-oxadiazole (2n) White solid, mp 148-152 °C; IR (KBr): 1211, 1289, 1545, 1603 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 3.88 (s, 6H), 6.63 (s, 1H), 7.27 (d, 2H, J = 1.5 Hz), 7.54-7.52 (m, 3H), 8.12 (d, 2H, J = 6.5 Hz).

2-(2-Chlorophenyl)-5-phenyl-1,3,4-oxadiazole (2o) White solid, mp 148-149 °C; IR (KBr): 799, 1019, 1091, 1260 1517 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.43-7.46 (m, 1H), 7.48-7.51 (m, 1H), 7.53-7.59 (m, 4H), 8.11 (d, 1H, J = 2.0 Hz), 8.13 (d, 2H, J = 1.5 Hz).

2-(3,5-Dimethoxyphenyl)-5-(4-methoxyphenyl)-1,3,4-oxadiazole (2p) White solid, mp 168-172 °C; IR (KBr): 1164, 1262, 1499, 1616 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 3.89 (s, 9H), 6.63 (s, 1H), 7.05 (dd, 2H, J = 3.0 Hz, J = 8.5 Hz), 7.26 (s, 2H), 8.08 (dd, 2H, J_1 = 3.0 Hz, J_2 = 8.5 Hz).

4-(5-(3,5-Dimethoxyphenyl)-1,3,4-oxadiazol-2-yl)-*N*,*N*-dimethylbenzenamine (2q) White solid, mp 218-220 °C; IR (KBr): 1194, 1262, 1506, 1614 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 3.08 (s, 6H), 3.89 (s, 6H), 6.62 (d, 1H, J = 1.0 Hz), 6.77 (d, 2H, J = 8.5 Hz), 7.27 (s, 2H), 7.98 (d, 2H, J = 8.5 Hz).

2-(3,5-Dimethoxyphenyl)-5-(3-nitrophenyl)-1,3,4-oxadiazole (2r) White solid, mp 279-281 °C; IR (KBr): 1159, 1210, 1349, 1527, 3075 cm^{-1}; ^{1}H-NMR (500 MHz, DMSO-d_6) δ 3.88 (s, 6H), 6.80 (s, 1H), 7.3 (s, 2H), 7.94 (t, 1H, J = 8.0 Hz), 8.49 (d, 1H, J = 8.5 Hz), 8.59 (d, 1H, J = 8.0 Hz), 8.84 (s, 1H).

2-(3,5-Dimethoxyphenyl)-5-(furan-2-yl)-1,3,4-oxadiazole (2s) White solid, mp 148-150 °C. IR (KBr): 1161, 1210, 1553, 1634 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$) δ 3.88 (s, 6H), 6.63 (s, 2H), 7.23 (d, 1H, J = 2.5 Hz), 7.26 (s, 2H), 7.67 (s, 1H).

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