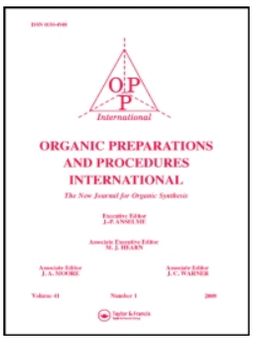

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Convenient Vilsmeier Reagent Mediated One-Pot Synthesis of Symmetrical and Asymmetrical 1,3,4-Oxadiazoles

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The 1,3,4-oxadiazole structure is considered to be a privileged scaffold in medicinal and pesticide chemistry.[1] These important five-membered heterocycles have diverse biological properties such as antibacterial, antifungal, antitubercular, insecticidal, herbicidal, analgesic, anti-inflammatory, anticonvulsant and anticancer activities.[2] Drugs displaying a 1,3,4-oxadiazole ring include an antiretroviral drug for the treatment of HIV infection (raltegravir), an anticancer agent (zibotentan) and an antihypertensive (nesapidil) (*Figure 1*).

1,3,4-Oxadiazoles have also shown wide applications in material science.[3] In line with their importance, there are numerous methods available for the construction of the 1,3,4-oxadiazole ring.[4] A widely used method is dehydrative cyclization of diacylhydrazines. Because of harsh dehydration reagents, such as H_2SO_4,[5] $POCl_3$[6–7] and polyphosphoric acid (PPA)[8], some effort has been made to develop modified and milder procedures.

1-Ethyl-3-(3-dimethylaminopropyl) carbodiimide,[9] Ph_3PO and Tf_2O,[10] propylphosphonic anhydride (T3P),[11] XtalFluor-E,[12] EDCI[13] and Burgess reagent[14] were reported in the literature for cyclodehydration of diacylhydrazines to 1,3,4-oxadiazoles. The use of hazardous materials, high cost, low yield of products, tedious separation of products and high temperature are the major limitations associated with these procedures. Thus the development of new and efficient methodologies for synthesis of 1,3,4-oxadiazoles is important.

Chloromethylenedimethylammonium chloride (Vilsmeier reagent) **1** has mostly been used as a formylating agent.[15] It was also applied as a powerful water scavenger and coupling reagent for the synthesis of esters,[16] amides,[17] acid chlorides,[18] β-sultams[19] and β-lactams.[20–22] Preparation of this reagent is easy from DMF and oxalyl chloride or thionyl chloride in dry dichloromethane.[23] High safety and ease of handling of this reagent are its other advantages. Herein, we report a convenient one-pot synthesis of 1,3,4-oxadiazoles by employing Vilsmeier reagent as acid activator and coupling reagent.

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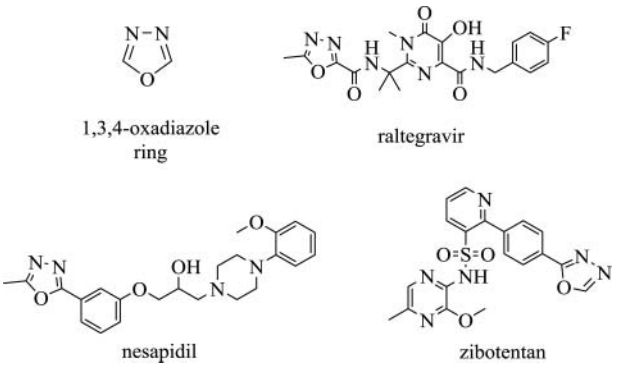
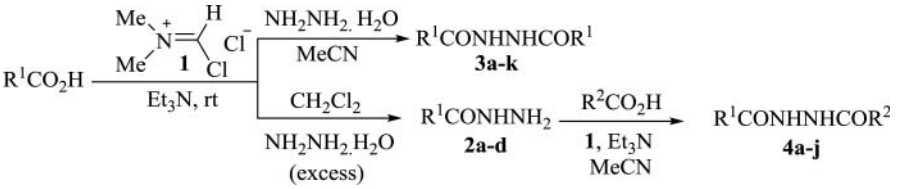


Figure 1 1,3,4-Oxadiazole ring and commercially 1,3,4-oxadiazole drugs.

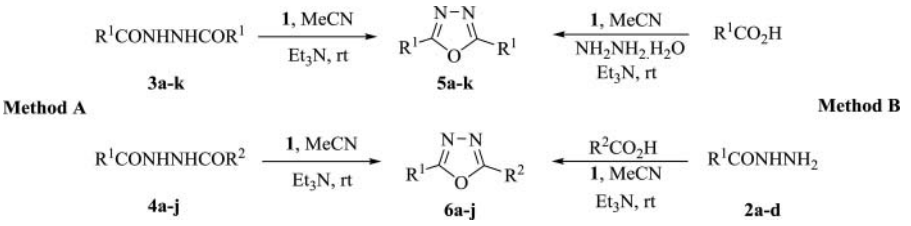
Recently, we reported simple protocols for the one-pot synthesis of acylhydrazines and symmetrical and asymmetrical diacylhydrazines from carboxylic acids using Vilsmeier reagent.[24] Reaction of carboxylic acids with excess hydrazine monohydrate (100%) in the presence of Vilsmeier reagent and triethylamine in dry CH_2Cl_2 at room temperature gave acylhydrazines **2a-d**. Symmetrical diacylhydrazines **3a-k** were directly prepared from the carboxylic acids *via* Vilsmeier reagent in dry acetonitrile. Asymmetrical diacylhydrazines **4a-j** were obtained by treatment of acylhydrazines **2a-d** with a solution of the corresponding carboxylic acids, triethylamine and reagent **1** in dry acetonitrile (*Scheme 1*).[24]



Scheme 1

The above successful results encouraged us to try the synthesis of 1,3,4-oxadiazoles from diacylhydrazines using Vilsmeier reagent as a cyclodehydration reagent. Our initial studies focused on the model reaction of diacylhydrazine **3a** ($R^1 = C_6H_5OCH_2$). Treatment of diacylhydrazine **3a** with trimethylamine (3.0 equiv) and Vilsmeier reagent (1.0 equiv) in dry acetonitrile at room temperature gave symmetrical 1,3,4-oxadiazole **5a** with 91% yield after crystallization from ethanol. The progress of reaction was checked by TLC monitoring. After completion of the reaction, water was added and extraction by ethyl acetate was performed.

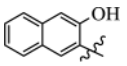
With these results in hand, the scope of the reaction was extended to a diverse range of 1,3,4-oxadiazoles. As can be seen from *Scheme 2* and *Table 1* (Method A), a wide range of symmetrical and asymmetrical diacylhydrazines containing aromatic and aliphatic substituents can all be converted into the corresponding 1,3,4-oxadiazoles in good to excellent yields and with high purity.



Scheme 2

Preparation of 1,3,4-oxadiazoles **5a-k** and **6a-j** in two and three stages from carboxylic acids using Vilsmeier reagent (Method A), respectively, persuaded us to try a direct one-pot synthesis of symmetrical 1,3,4-oxadiazoles **5a-k** from the carboxylic acids and asymmetrical 1,3,4-oxadiazoles **6a-j** from acylhydrazines **2a-d** (Method B). In this way, the reaction of phenoxyacetic acid (1.0 mmol) with hydrazine hydrate (0.3 mmol) in the presence of Vilsmeier reagent **1** (1.0 mmol) and triethylamine (1.0 mmol) in dry acetonitrile at room temperature overnight gave crude 1,3,4-oxadiazole **5a** (Method B). Saturated $NaHCO_3$ was added to the mixture and extraction with ethyl acetate was performed. Pure 1,3,4-oxadiazole **5a** was obtained after crystallization from ethanol with a nearly identical

Table 1
Synthesis of 1,3,4-oxadiazoles **5a-k** and **6a-j** Using Vilsmeier Reagent

				Isolated yield (%)	
Entry	R^1	R^2	Product	Method A	Method B
1	$C_6H_5OCH_2$	–	**5a**	91	89
2	2,4-$Cl_2C_6H_3OCH_2$	–	**5b**	92	88
3	4-$ClC_6H_4OCH_2$	–	**5c**	88	89
4	PhthN-CH_2	–	**5d**	85	90
5	2-NaphthOCH_2	–	**5e**	90	92
6		–	**5f**	93	90
7	3-OHC_6H_4	–	**5g**	83	85
8	C_6H_5	–	**5h**	89	84
9	2,6-$(MeO)_2C_6H_3$	–	**5i**	93	88
10	4-OHC_6H_4	–	**5j**	84	89
11	$MeOCH_2$	–	**5k**	86	87
12	$C_6H_5OCH_2$	4-$ClC_6H_4OCH_2$	**6a**	86	85
13	$C_6H_5OCH_2$	$MeOCH_2$	**6b**	81	84
14	2,4-$Cl_2C_6H_3OCH_2$	$C_6H_5OCH_2$	**6c**	80	82
15	2,4-$Cl_2C_6H_3OCH_2$	4-$ClC_6H_4OCH_2$	**6d**	82	86
16	2,4-$Cl_2C_6H_3OCH_2$	$MeOCH_2$	**6e**	81	89
17	2-NaphthOCH_2	$C_6H_5OCH_2$	**6f**	84	80
18	2-NaphthOCH_2	4-$ClC_6H_4OCH_2$	**6g**	87	85
19	2-NaphthOCH_2	2,4-$Cl_2C_6H_3OCH_2$	**6h**	95	93
20	2-NaphthOCH_2	$MeOCH_2$	**6i**	88	85
21	4-$ClC_6H_4OCH_2$	$MeOCH_2$	**6j**	84	87

yield with Method A. Pure asymmetrical 1,3,4-oxadiazoles **6a-j** were also prepared directly from acylhydrazines **2a-d** (*Scheme 2*, Method B, *Table 1*). The synthesized compounds were characterized by IR, ^{1}H NMR, ^{13}C NMR and elemental analysis. The IR spectra showed the C = N peak at 1604–1627 cm^{-1} and the disappearance of the NH and CO peaks of diacylhydrazines. Furthermore, the ^{13}C NMR spectra showed the C = N peaks at 154.8–165.3 ppm.

In conclusion, efficient methods for a convenient preparation of symmetrical and asymmetrical 1,3,4-oxadiazoles have been developed through Vilsmeier reagent-mediated reactions of hydrazine hydrate with diverse carboxylic acids. The method is simple and the easy workup removes by-products. Pure products are obtained by crystallization from ethanol without the need for column chromatography. In addition, this reagent offers advantages over the existing ones by creating milder reaction conditions.

Experimental Section

All required chemicals were purchased from Merck, Aldrich and Acros chemical companies. The melting points were determined on a silicone oil bath and are uncorrected. IR spectra were measured on a Galaxy series FT-IR 5000 spectrometer. NMR spectra were recorded in DMSO-d_6 using a Bruker spectrophotometer (^{1}H NMR 300 MHz, ^{13}C NMR 75 MHz) with tetramethylsilane as an internal standard and coupling constants were given in cycles per second (Hz). Elemental analyses were run on a Vario EL III elemental analyzer. Thin-layer chromatography was carried out on silica gel 254 analytical sheets obtained from Fluka. (Chloromethylene)dimethylammonium chloride (Vilsmeier reagent) **1** was obtained as a white solid by a reported procedure.[23] Procedures for the synthesis of acylhydrazines **2a-d**, diacylhydrazines **3a-k**, **4a-j** and their spectral data have been previously reported.[24] All solvents and triethylamine were dried prior to use according to standard methods [25] and then stored over molecular sieves (4Å). All glassware was dried by hot air, and reactions were carried out under a $CaCl_2$-tube to prevent the entry of moisture into the reaction vessel.

General Procedure for the Synthesis of Symmetrical and Asymmetrical 1,3,4-oxadiazoles 5a-k and 6a-j (Method A)

Symmetrical diacylhydrazines **3a-k** and asymmetrical diacylhydrazines **4a-j** were converted to symmetrical and asymmetrical 1,3,4-oxadiazoles **5a-k** and **6a-j**, respectively, by this method. A solution of diacylhydrazine (1.0 mmol), Vilsmeier reagent (1.0 mmol) and Et_3N (3.0 mmol) in dry CH_3CN (20 mL) was stirred at room temperature for 8 hours. Water (20 mL) was added and the mixture was extracted with EtOAc (3 × 20 mL). The organic layer was washed with brine (20 mL), dried (Na_2SO_4), filtered and the solvent was removed under reduced pressure to give the crude products. The crude residues were purified by crystallization from 95% ethanol.

General Procedure for One-pot Synthesis of Symmetrical 1,3,4-oxadiazoles 5a-k (Method B)

Hydrazine hydrate (0.3 mmol) was added to a solution of carboxylic acid (1.0 mmol), Vilsmeier reagent (1.0 mmol) and Et_3N (2.0 mmol) in dry CH_3CN (20 mL) at room temperature. After 2 hours Vilsmeier reagent (0.4 mmol) and Et_3N (1.0 mmol) were added and the mixture was stirred at room temperature for 8 hours. Saturated $NaHCO_3$ (20 mL)

was added and the mixture was extracted with EtOAc (3 × 20 mL). The organic layer was washed with brine (20 mL), dried (Na_2SO_4), filtered and the solvent was removed under reduced pressure to give the crude products. The crude residues were purified by crystallization from 95% ethanol. Spectral data for **5a-b**, **5d**, **5f-h** and **5j** have been previously reported,[26–32] and were matched by the products of this work.

2,5-bis((4-Chlorophenoxy)methyl)-1,3,4-oxadiazole (5c)

White solid. mp: 108–110°C. IR (KBr) cm^{-1}: 1620 (C = N). ^1H NMR δ 5.20 (2 CH_2, s, 4H), 6.81–7.30 (ArH, m, 8H); ^{13}C NMR δ 57.1 (CH_2), 116.0, 127.1, 129.3, 157.1 (aromatic carbons), 157.2 (C = N).

Anal. Calcd for $C_{16}H_{12}Cl_2N_2O_3$: C, 54.72; H, 3.44; N, 7.98. Found: C, 54.79; H, 3.53; N, 8.02.

2,5-bis((Naphthalen-2-yloxy)methyl)-1,3,4-oxadiazole (5e)

White solid. Mp: 249–251°C. IR (KBr) cm^{-1}: 1619 (C = N). ^1H NMR δ 5.19 (2 CH_2, s, 4H), 7.04–7.76 (ArH, m, 14H); ^{13}C NMR δ 57.2 (CH_2), 109.1, 117.6, 124.0, 127.2, 127.3, 128.7, 129.4, 131.1, 135.1, 156.1 (aromatic carbons), 157.1 (C = N).

Anal. Calcd for $C_{24}H_{18}N_2O_3$: C, 75.38; H, 4.74; N, 7.33. Found: C, 75.31; H, 4.68; N, 7.30.

2,5-bis(2,6-Dimethoxyphenyl)-1,3,4-oxadiazole (5i)

Pale-yellow solid. mp: 152–154°C. IR (KBr) cm^{-1}: 1627 (C = N). ^1H NMR δ 3.78 (4 OMe, s, 12H), 6.70–7.34 (ArH, m, 6H); ^{13}C NMR δ 56.4 (OMe), 108.8, 111.0, 131.4, 155.7 (aromatic carbons), 161.4 (C = N).

Anal. Calcd for $C_{18}H_{18}N_2O_5$: C, 63.15; H, 5.30; N, 8.18. Found: C, 63.29; H, 5.47; N, 8.27.

2,5-bis(methoxymethyl)-1,3,4-oxadiazole (5k)

White solid. mp 144–146°C. IR (KBr) cm^{-1}: 1609 (C = N). ^1H NMR δ 3.37 (2 OMe, s, 6H), 4.42 (2 CH_2, s, 4H); ^{13}C NMR δ 58.8 (OMe), 60.3 (CH_2), 165.3 (C = N).

Anal. Calcd for $C_6H_{10}N_2O_3$: C, 45.57; H, 6.37; N, 17.71. Found: C, 45.69; H, 6.51; N, 17.77.

General Procedure for One-pot Synthesis of Asymmetrical 1,3,4-oxadiazoles 6a-j (Method B)

Acylhydrazines **2a-d** (1.0 mmol) were added to a solution of the appropriate carboxylic acid (1.2 mmol), Vilsmeier reagent (1.2 mmol) and Et_3N (2.5 mmol) in dry CH_3CN (20 mL) at room temperature. After 2 hours Vilsmeier reagent (1.0 mmol) and Et_3N (2.5 mmol) were added and the mixture was stirred at room temperature 8 hours. Saturated $NaHCO_3$ (20 mL) was added and the mixture was extracted with EtOAc (3 × 20 mL). The organic layer was washed with brine (20 mL), dried (Na_2SO_4), filtered and the solvent was removed under reduced pressure to give the crude products. The crude residues were purified by crystallization from 95% ethanol. The data for **6f** have been previously reported,[33] and matched those from this work.

2-((4-Chlorophenoxy)methyl)-5-(phenoxymethyl)-1,3,4-oxadiazole (6a)

White solid. mp: 224–226°C. IR (KBr) cm^{-1}: 1622 (C = N). ^1H NMR δ 5.18, 5.19 (2 CH_2, 2s, 4H), 6.82–7.28 (ArH, m, 9H); ^{13}C NMR δ 58.5, 61.7 (CH_2), 115.5, 115.9, 122.2, 126.9, 129.1, 129.8, 157.2, 157.3 (aromatic carbons), 158.8, 163.9 (C = N).

Anal. Calcd for $C_{16}H_{13}ClN_2O_3$: C, 60.67; H, 4.14; N, 8.84. Found: C, 60.75; H, 4.28; N, 8.90.

2-(Methoxymethyl)-5-(phenoxymethyl)-1,3,4-oxadiazole (6b)

Silver solid. mp: 208–210°C. IR (KBr) cm^{-1}: 1610 (C = N). ^1H NMR δ 3.30 (OMe, s, 3H), 4.47, 5.18 (2 CH_2, 2s, 4H), 6.89–7.27 (ArH, m, 5H); ^{13}C NMR δ 56.6 (OMe), 58.9, 60.7 (CH_2), 116.2, 120.7, 129.7, 157.4 (aromatic carbons), 159.6, 165.0 (C = N).

Anal. Calcd for $C_{11}H_{12}N_2O_3$: C, 59.99; H, 5.49; N, 12.72. Found: C, 60.12; H, 5.64; N, 12.66.

2-((2,4-Dichlorophenoxy)methyl)-5-(phenoxymethyl)-1,3,4-oxadiazole (6c)

White solid. mp: 148–150°C. IR (KBr) cm^{-1}: 1621 (C = N). ^1H NMR δ 5.09, 5.19 (2 CH_2, 2s, 4H), 6.87–7.39 (ArH, m, 8H); ^{13}C NMR δ 57.4, 58.2 (CH_2), 115.6, 118.6, 121.7, 125.5, 128.6, 128.7, 129.7, 130.5, 138.1, 153.7 (aromatic carbons), 157.3, 159.3 (C = N).

Anal. Calcd for $C_{16}H_{12}Cl_2N_2O_3$: C, 54.72; H, 3.44; N, 7.98. Found: C, 54.66; H, 3.53; N, 7.94.

2-((4-Chlorophenoxy)methyl)-5-((2,4-dichlorophenoxy) methyl)-1,3,4-oxadiazole (6d)

White solid. mp: 257–259°C. IR (KBr) cm^{-1}: 1613 (C = N). ^1H NMR δ 5.08, 5.19 (2 CH_2, 2s, 4H), 6.82–7.39 (ArH, m, 7H); ^{13}C NMR δ 56.6, 57.3 (CH_2), 115.4, 117.4, 125.8, 127.1, 128.4, 128.7, 129.3, 131.1, 148.4, 153.6 (aromatic carbons), 154.8, 157.3 (C = N).

Anal. Calcd for $C_{16}H_{11}Cl_3N_2O_3$: C, 49.83; H, 2.88; N, 7.26. Found: C, 49.88; H, 2.97; N, 7.31.

2-((2,4-Dichlorophenoxy)methyl)-5-(methoxymethyl)-1,3,4-oxadiazole (6e)

White solid. mp: 160–162°C. IR (KBr) cm^{-1}: 1612 (C = N). ^1H NMR δ 3.43 (OMe, s, 3H), 4.46, 5.13 (2 CH_2, 2s, 4H), 6.91–7.41 (ArH, m, 3H); ^{13}C NMR δ 57.1 (OMe), 58.0, 60.6 (CH_2), 117.6, 125.9, 128.2, 128.8, 131.7, 152.6 (aromatic carbons), 158.2, 165.2 (C = N).

Anal. Calcd for $C_{11}H_{10}Cl_2N_2O_3$: C, 45.70; H, 3.49; N, 9.69. Found: C, 45.82; H, 3.65; N, 9.61.

2-((4-Chlorophenoxy)methyl)-5-((naphthalen-2-yloxy)methyl)-1,3,4-oxadiazole (6g)

White solid. mp: 170–172°C. IR (KBr) cm^{-1}: 1623 (C = N). ^1H NMR δ 5.04, 5.15 (2 CH_2, 2s, 4H), 6.82–7.78 (ArH, m, 11H); ^{13}C NMR δ 53.3, 57.1 (CH_2), 109.7, 116.0, 117.7, 123.4, 127.1, 127.2, 127.6, 128.4, 129.5, 129.6, 131.1, 135.5, 150.3, 156.4 (aromatic carbons), 157.5, 157.6 (C = N).

Anal. Calcd for $C_{20}H_{15}ClN_2O_3$: C, 65.49; H, 4.12; N, 7.64. Found: C, 65.41; H, 4.06; N, 7.59.

2-((2,4-Dichlorophenoxy)methyl)-5-((naphthalen-2-yloxy)methyl)-1,3,4-oxadiazole (6h)

Off-white solid. mp: 187–189°C. IR (KBr) cm^{-1}: 1611 (C = N). ^{1}H NMR δ 4.93, 5.01 (2 CH_2, 2s, 4H), 6.80–7.72 (ArH, m, 10H); ^{13}C NMR δ 58.0, 58.2 (CH_2), 108.0, 117.5, 117.9, 124.5, 125.8, 127.0, 127.4, 128.0, 128.5, 128.8, 129.7, 130.1, 132.5, 135.5, 147.5, 153.7 (aromatic carbons), 157.0, 157.4 (C = N).

Anal. Calcd for $C_{20}H_{14}Cl_2N_2O_3$: C, 59.87; H, 3.52; N, 6.98. Found: C, 59.99; H, 3.66; N, 7.06.

2-(Methoxymethyl)-5-((naphthalen-2-yloxy)methyl)-1,3,4-oxadiazole (6i)

White solid. mp: 176–178°C. IR (KBr) cm^{-1}: 1604 (C = N). ^{1}H NMR δ 3.32 (OMe, s, 3H), 4.45, 5.13 (2 CH_2, 2s, 4H), 7.09–7.78 (ArH, m, 7H); ^{13}C NMR δ 56.5 (OMe), 57.7, 60.6 (CH_2), 109.1, 117.3, 124.2, 127.1, 127.5, 128.5, 129.4, 132.2, 136.0, 156.2 (aromatic carbons), 157.3, 165.0 (C = N).

Anal. Calcd for $C_{15}H_{14}N_2O_3$: C, 66.66; H, 5.22; N, 10.36. Found: C, 66.58; H, 5.31; N, 10.33.

2-((4-Chlorophenoxy)methyl)-5-(methoxymethyl)-1,3,4-oxadiazole (6j)

Cream-colour solid. mp: 213–215°C. IR (KBr) cm^{-1}: 1616 (C = N). ^{1}H NMR δ 3.39 (OMe, s, 3H), 4.41, 5.01 (2 CH_2, 2s, 4H), 6.84 (ArH, d, 2H, J = 7.4), 7.28 (ArH, d, 2H, J = 7.4); ^{13}C NMR δ 57.6 (OMe), 58.9, 60.9 (CH_2), 116.1, 127.5, 129.5, 157.4 (aromatic carbons), 157.9, 164.7 (C = N).

Anal. Calcd for $C_{11}H_{11}ClN_2O_3$: C, 51.88; H, 4.35; N, 11.00. Found: C, 51.97; H, 4.48; N, 11.07.

Acknowledgments

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