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# 5-Chloromethyl-3-(3,5-dimethoxyphenyl)-1,2,4-oxadiazole

**Pin-liang Wang, Hai-su Zeng, Hai-bo Wang* and Wen-yuan Wu**

College of Science, Nanjing University of Technology, Xinmofan Road No. 5 Nanjing, Nanjing 210009, People's Republic of China
Correspondence e-mail: wanghaibo@njut.edu.cn

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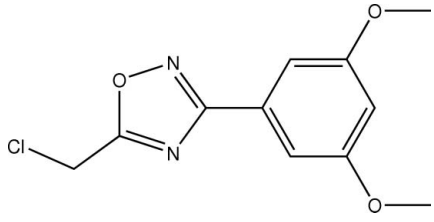
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.005 Å; $R$ factor = 0.054; $wR$ factor = 0.173; data-to-parameter ratio = 14.6.

In the title compound, $C_{11}H_{11}ClN_2O_3$, the substituted phenyl ring and the oxadiazole system are essentially coplanar. Close intramolecular C—H···N interactions are observed.

## Related literature

For related literature, see: Nicolaides *et al.* (1998); Romero (2001); Terashita *et al.* (2002).




## Experimental

### Crystal data

$C_{11}H_{11}ClN_2O_3$
$M_r$ = 254.67
Triclinic, $P\overline{1}$
$a$ = 7.861 (2) Å
$b$ = 8.502 (2) Å
$c$ = 9.537 (2) Å
$\alpha$ = 80.72 (3)°
$\beta$ = 73.90 (3)°
$\gamma$ = 70.09 (3)°
$V$ = 574.2 (2) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.33 mm$^{-1}$
$T$ = 293 (2) K
0.40 × 0.20 × 0.20 mm

### Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: $\psi$ scan (North *et al.*, 1968)
$T_{min}$ = 0.879, $T_{max}$ = 0.937
2422 measured reflections
2249 independent reflections
1589 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.031
3 standard reflections
every 200 reflections
intensity decay: none

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.173
$S$ = 1.08
2249 reflections
154 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.34 e Å$^{-3}$

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C9—H9$A$···N2 | 0.93 | 2.60 | 2.925 (5) | 101 |
| C5—H5$A$···N1 | 0.93 | 2.54 | 2.838 (5) | 99 |

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1996); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2037).

## References

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# supplementary materials

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# 5-Chloromethyl-3-(3,5-dimethoxyphenyl)-1,2,4-oxadiazole

**P. Wang, H. Zeng, H. Wang and W. Wu**

## Comment

1,2,4-Oxadiazoles are an important class of five-membered heterocyclic compounds. Some derivatives of 1,2,4-oxadiazoles showed intrinsic analgesic (Terashita *et al.*, 2002), antiinflammatory (Nicolaides *et al.*, 1998) and antipicornaviral (Romero, 2001) properties. We report here the crystal structure of the title compound, (I). The molecular structure of (I) is shown in Fig. 1, where the dashed lines indicate close C—H···N contacts which most probably are caused by the coplanar arrangement of the phenyl group and the oxadiazole system (Table 1). The other bond lengths and angles are of expected values (*cf.* Supplementary Material).

## Experimental

A solution of chloroacetylchloride (16 mmol) in toluene (10 ml) was added dropwise to a solution of 3,5-(Dimethoxyl)benzamidoxime (14 mmol) in toluene (60 ml). The resulting mixture was refluxed for 6 h, then concentrated under reduced pressure to afford crude compound(I). Pure compound (I) was obtained by recrystallization from a mixture of ethyl acetate (15 ml) and light petrolum (b.p. 60–90°C, 7.5 ml). Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution. $^1$H NMR ($CDCl_3$, δ, p.p.m.): 6.55–6.56 (m, 2H), 6.23–6.25 (m, 1H), 4.64 (s, 2H), 3.77 (s, 6H).

## Refinement

All H atoms bonded to the C atoms were placed geometrically at distances of 0.93–0.97 Å and included in the refinement using an riding motion approximation with $U_{iso}$(H) = 1.2 or 1.5$U_{eq}$ of the carrier atom.

## Figures

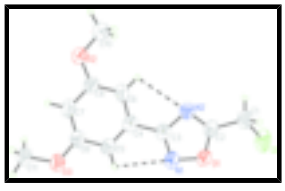

Fig. 1. A view of the molecular structure of (I), showing displacement ellipsoids at the 30% probability level. Dashed lines indicate C—H···N interactions.

## 5-Chloromethyl-3-(3,5-dimethoxyphenyl)-1,2,4-oxadiazole

*Crystal data*

| | |
|---|---|
| $C_{11}H_{11}ClN_2O_3$ | $Z = 2$ |
| $M_r = 254.67$ | $F_{000} = 264$ |
| Triclinic, $P\overline{1}$ | $D_x = 1.473$ Mg m$^{-3}$ |

| | |
|---|---|
| Hall symbol: -P 1 | Mo $K\alpha$ radiation<br>$\lambda$ = 0.71073 Å |
| $a$ = 7.861 (2) Å | Cell parameters from 25 reflections |
| $b$ = 8.502 (2) Å | $\theta$ = 9–13º |
| $c$ = 9.537 (2) Å | $\mu$ = 0.33 mm$^{-1}$ |
| $\alpha$ = 80.72 (3)º | $T$ = 293 (2) K |
| $\beta$ = 73.90 (3)º | Block, colourless |
| $\gamma$ = 70.09 (3)º | 0.40 × 0.20 × 0.20 mm |
| $V$ = 574.2 (2) Å$^3$ | |

*Data collection*

| | |
|---|---|
| Enraf–Nonius CAD-4 diffractometer | $R_{int}$ = 0.031 |
| Radiation source: fine-focus sealed tube | $\theta_{max}$ = 26.0º |
| Monochromator: graphite | $\theta_{min}$ = 2.2º |
| $T$ = 293(2) K | $h$ = −9→9 |
| $\omega/2\theta$ scans | $k$ = −10→10 |
| Absorption correction: $\psi$ scan (North *et al.*, 1968) | $l$ = 0→11 |
| $T_{min}$ = 0.879, $T_{max}$ = 0.937 | 3 standard reflections |
| 2422 measured reflections | every 200 reflections |
| 2249 independent reflections | intensity decay: none |
| 1589 reflections with $I > 2\sigma(I)$ | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)]$ = 0.054 | H-atom parameters constrained |
| $wR(F^2)$ = 0.173 | $w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S$ = 1.08 | $(\Delta/\sigma)_{max} < 0.001$ |
| 2249 reflections | $\Delta\rho_{max}$ = 0.31 e Å$^{-3}$ |
| 154 parameters | $\Delta\rho_{min}$ = −0.34 e Å$^{-3}$ |
| Primary atom site location: structure-invariant direct methods | Extinction correction: none |

*Special details*

**Geometry**. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted $R$-factor $wR$ and goodness of fit S are based on $F^2$, conventional $R$-factors $R$ are based on F, with F set to zero for negative $F^2$. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculat-

ing *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on $F^2$ are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Cl | 0.3102 (2) | 0.49956 (16) | 0.15386 (12) | 0.0816 (5) |
| O1 | 0.3892 (4) | 0.1611 (3) | 0.3056 (3) | 0.0548 (7) |
| O2 | −0.1673 (4) | −0.1007 (4) | 0.9489 (3) | 0.0684 (9) |
| O3 | 0.3152 (4) | −0.5222 (3) | 0.6710 (3) | 0.0578 (7) |
| N1 | 0.3733 (5) | 0.0065 (4) | 0.3778 (4) | 0.0538 (8) |
| N2 | 0.1641 (4) | 0.2229 (4) | 0.5042 (3) | 0.0451 (7) |
| C1 | 0.2542 (6) | 0.4563 (5) | 0.3446 (4) | 0.0577 (10) |
| H1B | 0.3402 | 0.4823 | 0.3861 | 0.069* |
| H1C | 0.1297 | 0.5283 | 0.3852 | 0.069* |
| C2 | 0.2623 (5) | 0.2789 (5) | 0.3874 (4) | 0.0448 (8) |
| C3 | 0.2402 (5) | 0.0511 (4) | 0.4938 (4) | 0.0414 (8) |
| C4 | 0.1792 (5) | −0.0743 (5) | 0.6045 (4) | 0.0425 (8) |
| C5 | 0.2710 (5) | −0.2428 (4) | 0.5881 (4) | 0.0444 (8) |
| H5A | 0.3678 | −0.2777 | 0.5065 | 0.053* |
| C6 | 0.2172 (5) | −0.3588 (4) | 0.6944 (4) | 0.0446 (8) |
| C7 | 0.0694 (5) | −0.3066 (5) | 0.8134 (4) | 0.0492 (9) |
| H7A | 0.0335 | −0.3858 | 0.8841 | 0.059* |
| C8 | −0.0273 (5) | −0.1346 (5) | 0.8283 (4) | 0.0478 (9) |
| C9 | 0.0298 (5) | −0.0180 (5) | 0.7237 (4) | 0.0469 (9) |
| H9A | −0.0304 | 0.0961 | 0.7325 | 0.056* |
| C10 | −0.2766 (6) | 0.0704 (5) | 0.9669 (5) | 0.0689 (12) |
| H10A | −0.3717 | 0.0780 | 1.0563 | 0.103* |
| H10B | −0.3335 | 0.1144 | 0.8859 | 0.103* |
| H10C | −0.1981 | 0.1342 | 0.9707 | 0.103* |
| C11 | 0.2735 (6) | −0.6468 (5) | 0.7836 (4) | 0.0576 (10) |
| H11A | 0.3507 | −0.7563 | 0.7535 | 0.086* |
| H11B | 0.1447 | −0.6389 | 0.8003 | 0.086* |
| H11C | 0.2970 | −0.6286 | 0.8722 | 0.086* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl | 0.1243 (11) | 0.0765 (8) | 0.0512 (6) | −0.0560 (8) | −0.0131 (6) | 0.0155 (5) |
| O1 | 0.0591 (16) | 0.0518 (16) | 0.0486 (15) | −0.0255 (13) | 0.0038 (12) | −0.0016 (12) |
| O2 | 0.083 (2) | 0.0516 (17) | 0.0531 (17) | −0.0204 (15) | 0.0089 (15) | −0.0010 (13) |
| O3 | 0.0741 (19) | 0.0412 (15) | 0.0502 (15) | −0.0192 (13) | −0.0034 (13) | 0.0006 (12) |
| N1 | 0.060 (2) | 0.0454 (18) | 0.0511 (19) | −0.0211 (15) | −0.0039 (16) | 0.0029 (14) |
| N2 | 0.0504 (18) | 0.0469 (18) | 0.0389 (16) | −0.0189 (14) | −0.0094 (13) | −0.0001 (13) |
| C1 | 0.070 (3) | 0.051 (2) | 0.051 (2) | −0.024 (2) | −0.012 (2) | 0.0041 (18) |
| C2 | 0.047 (2) | 0.051 (2) | 0.0414 (19) | −0.0225 (17) | −0.0130 (16) | 0.0006 (16) |
| C3 | 0.0431 (19) | 0.045 (2) | 0.0422 (19) | −0.0196 (15) | −0.0157 (15) | 0.0013 (15) |
| C4 | 0.0450 (19) | 0.051 (2) | 0.0402 (18) | −0.0238 (16) | −0.0155 (15) | 0.0024 (15) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C5 | 0.047 (2) | 0.050 (2) | 0.0375 (18) | −0.0187 (16) | −0.0077 (15) | −0.0029 (15) |
| C6 | 0.055 (2) | 0.042 (2) | 0.0414 (19) | −0.0190 (16) | −0.0166 (16) | 0.0010 (15) |
| C7 | 0.063 (2) | 0.046 (2) | 0.042 (2) | −0.0247 (18) | −0.0132 (18) | 0.0063 (16) |
| C8 | 0.056 (2) | 0.050 (2) | 0.0375 (19) | −0.0200 (17) | −0.0089 (16) | −0.0004 (15) |
| C9 | 0.058 (2) | 0.0422 (19) | 0.046 (2) | −0.0206 (17) | −0.0179 (17) | 0.0043 (15) |
| C10 | 0.076 (3) | 0.058 (3) | 0.061 (3) | −0.019 (2) | −0.002 (2) | −0.006 (2) |
| C11 | 0.072 (3) | 0.041 (2) | 0.052 (2) | −0.0175 (19) | −0.006 (2) | 0.0038 (17) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| Cl—C1 | 1.757 (4) | C4—C9 | 1.397 (5) |
| O1—C2 | 1.322 (4) | C5—C6 | 1.380 (5) |
| O1—N1 | 1.411 (4) | C5—H5A | 0.9300 |
| O2—C8 | 1.343 (4) | C6—C7 | 1.384 (5) |
| O2—C10 | 1.426 (5) | C7—C8 | 1.410 (5) |
| O3—C6 | 1.360 (4) | C7—H7A | 0.9300 |
| O3—C11 | 1.434 (4) | C8—C9 | 1.384 (5) |
| N1—C3 | 1.298 (5) | C9—H9A | 0.9300 |
| N2—C2 | 1.290 (4) | C10—H10A | 0.9600 |
| N2—C3 | 1.386 (4) | C10—H10B | 0.9600 |
| C1—C2 | 1.482 (5) | C10—H10C | 0.9600 |
| C1—H1B | 0.9700 | C11—H11A | 0.9600 |
| C1—H1C | 0.9700 | C11—H11B | 0.9600 |
| C3—C4 | 1.478 (5) | C11—H11C | 0.9600 |
| C4—C5 | 1.380 (5) | | |
| C2—O1—N1 | 106.2 (3) | O3—C6—C7 | 124.1 (3) |
| C8—O2—C10 | 117.5 (3) | C5—C6—C7 | 120.3 (3) |
| C6—O3—C11 | 117.6 (3) | C6—C7—C8 | 120.6 (3) |
| C3—N1—O1 | 103.1 (3) | C6—C7—H7A | 119.7 |
| C2—N2—C3 | 101.7 (3) | C8—C7—H7A | 119.7 |
| C2—C1—Cl | 112.7 (3) | O2—C8—C9 | 126.2 (4) |
| C2—C1—H1B | 109.1 | O2—C8—C7 | 114.7 (3) |
| Cl—C1—H1B | 109.1 | C9—C8—C7 | 119.1 (3) |
| C2—C1—H1C | 109.1 | C8—C9—C4 | 119.1 (3) |
| Cl—C1—H1C | 109.1 | C8—C9—H9A | 120.5 |
| H1B—C1—H1C | 107.8 | C4—C9—H9A | 120.5 |
| N2—C2—O1 | 114.5 (3) | O2—C10—H10A | 109.5 |
| N2—C2—C1 | 126.6 (3) | O2—C10—H10B | 109.5 |
| O1—C2—C1 | 118.8 (3) | H10A—C10—H10B | 109.5 |
| N1—C3—N2 | 114.5 (3) | O2—C10—H10C | 109.5 |
| N1—C3—C4 | 121.6 (3) | H10A—C10—H10C | 109.5 |
| N2—C3—C4 | 123.9 (3) | H10B—C10—H10C | 109.5 |
| C5—C4—C9 | 121.8 (3) | O3—C11—H11A | 109.5 |
| C5—C4—C3 | 119.4 (3) | O3—C11—H11B | 109.5 |
| C9—C4—C3 | 118.7 (3) | H11A—C11—H11B | 109.5 |
| C6—C5—C4 | 119.1 (3) | O3—C11—H11C | 109.5 |
| C6—C5—H5A | 120.5 | H11A—C11—H11C | 109.5 |
| C4—C5—H5A | 120.5 | H11B—C11—H11C | 109.5 |
| O3—C6—C5 | 115.6 (3) | | |

| | | | |
|---|---|---|---|
| C2—O1—N1—C3 | −0.4 (4) | C3—C4—C5—C6 | 177.8 (3) |
| C3—N2—C2—O1 | 0.6 (4) | C11—O3—C6—C5 | 175.6 (3) |
| C3—N2—C2—C1 | −174.9 (4) | C11—O3—C6—C7 | −6.0 (5) |
| N1—O1—C2—N2 | −0.1 (4) | C4—C5—C6—O3 | −179.5 (3) |
| N1—O1—C2—C1 | 175.7 (3) | C4—C5—C6—C7 | 2.0 (5) |
| Cl—C1—C2—N2 | −150.1 (3) | O3—C6—C7—C8 | −178.9 (3) |
| Cl—C1—C2—O1 | 34.6 (5) | C5—C6—C7—C8 | −0.5 (6) |
| O1—N1—C3—N2 | 0.8 (4) | C10—O2—C8—C9 | 4.6 (6) |
| O1—N1—C3—C4 | −178.7 (3) | C10—O2—C8—C7 | −177.0 (4) |
| C2—N2—C3—N1 | −0.9 (4) | C6—C7—C8—O2 | −179.7 (4) |
| C2—N2—C3—C4 | 178.6 (3) | C6—C7—C8—C9 | −1.2 (6) |
| N1—C3—C4—C5 | 3.0 (5) | O2—C8—C9—C4 | 179.7 (4) |
| N2—C3—C4—C5 | −176.4 (3) | C7—C8—C9—C4 | 1.3 (5) |
| N1—C3—C4—C9 | −177.3 (4) | C5—C4—C9—C8 | 0.2 (5) |
| N2—C3—C4—C9 | 3.3 (5) | C3—C4—C9—C8 | −179.5 (3) |
| C9—C4—C5—C6 | −1.9 (5) | | |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C9—H9A···N2 | 0.93 | 2.60 | 2.925 (5) | 101 |
| C5—H5A···N1 | 0.93 | 2.54 | 2.838 (5) | 99 |

**Fig. 1**