5-Chloromethyl-3-(4-methylphenyl)-1,2,4-oxadiazole

Pin-Liang Wang, Hai-Su Zeng, Hai-Bo Wang* and Wen-Yuan Wu

College of Science, Nanjing University of Technolgy, Xinmofan Road No.5, Nanjing 210009, People's Republic of China

Correspondence e-mail: wanghaibo@njut.edu.cn

In the title compound, $C_{10}H_9ClN_2O$, the benzene and oxadiazole rings are roughly coplanar. The crystal packing shows weak intermolecular C—H⋯N interactions.

Related literature

For related literature, see: Juraszyk *et al.* (1997); Naka & Kubo (1999); Nicolaides *et al.* (1998); Romero (2001); Terashita *et al.* (2002).
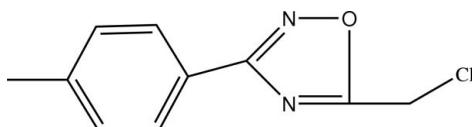
Experimental

Crystal data

$C_{10}H_9ClN_2O$	V = 1998.9 (7) Å^3
M_r = 208.64	Z = 8
Monoclinic, $C2/c$	Mo $K\alpha$ radiation
a = 25.462 (5) Å	μ = 0.35 mm^{-1}
b = 6.4040 (13) Å	T = 293 (2) K
c = 14.589 (3) Å	0.40 × 0.10 × 0.10 mm
β = 122.83 (3)°	

Data collection

Enraf–Nonius CAD-4 diffractometer	1959 independent reflections
Absorption correction: ψ scan (North *et al.*, 1968)	1259 reflections with $I > 2\sigma(I)$
	R_{int} = 0.041
T_{min} = 0.873, T_{max} = 0.966	3 standard reflections
2002 measured reflections	every 200 reflections
	intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057	127 parameters
$wR(F^2)$ = 0.182	H-atom parameters constrained
S = 1.02	$\Delta\rho_{max}$ = 0.28 e Å^{-3}
1959 reflections	$\Delta\rho_{min}$ = −0.31 e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C10—H10B⋯N1^i	0.97	2.52	3.436 (5)	157

Symmetry code: (i) $x, y - 1, z$.

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: KJ2072).

References

Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.

Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.

Juraszyk, H., Gante, J., Wurziger, H., Bernotat-Danielowski, S. & Melzer, G. (1997). Pct Int. Appl. WO 9 744 333.

Naka, T. & Kubo, K. (1999). *Curr. Pharm. Des.* **5**, 453–472.

Nicolaides, D. N., Fylaktakidou, K. C., Litinas, K. E. & Hadjipavlou-Litina, D. (1998). *Eur. J. Med. Chem.* **33**, 715–724.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Romero, J. R. (2001). *Exp. Opin. Invest. Drugs*, **10**, 369–379.

Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.

Siemens (1996). *SHELXTL*. Version 5.06. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Terashita, Z., Naruo, K. & Morimoto, S. (2002). Pct Int. Appl. WO 0 260 439.

supplementary materials

5-Chloromethyl-3-(4-methylphenyl)-1,2,4-oxadiazole

P.-L. Wang, H.-S. Zeng, H.-B. Wang and W.-Y. Wu

Comment

1,2,4-Oxadiazoles represent an important class of five-membered heterocycles. Some derivatives of 1,2,4-oxadiazoles have intrinsic analgesic (Terashita *et al.*, 2002), antiinflammatory (Nicolaides *et al.*, 1998) and antipicornaviral (Romero, 2001) properties and are efficient as agonists [*e.g.* forangiotensin (Naka *et al.*, 1999) and adhesion agents (Juraszyk *et al.*, 1997)] for different types of receptors, such as the peroxisome proliferator-activated receptor.

The molecular structure of the title compound is shown in Fig. 1. The structure analysis demonstrates that the benzene and oxadiazole rings are roughly coplanar, making a twist angle of only 4.4 (3). The crystal packing shows weak intermolecular C—H···N interactions.

Experimental

A solution of chloroacetylchloride (12 mmol) in toluene (10 ml) was added dropwise to a solution of 4-methylbenzamidoxime (10 mmol) in toluene (60 ml). The resulting mixture was refluxed for 6 h. After cooling and filtrating, the crude title compound was gained. The pure title compound was obtained by recrystallizing from a mixture of ethyl acetate (8 ml) and petrolum ether (7.5 ml). Crystals suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution.

Refinement

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

Figures
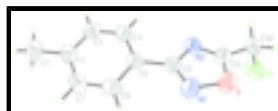
Fig. 1. A view of the molecular structure of the title compound, showing displacement ellipsoids at the 30% probability level.

5-Chloromethyl-3-(4-methylphenyl)-1,2,4-oxadiazole

Crystal data

$C_{10}H_9ClN_2O$	$F_{000} = 864$
$M_r = 208.64$	$D_x = 1.387$ Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -C 2yc	Cell parameters from 25 reflections
$a = 25.462$ (5) Å	$\theta = 9–13°$

$b = 6.4040\ (13)$ Å

$c = 14.589\ (3)$ Å

$\beta = 122.83\ (3)°$

$V = 1998.9\ (7)$ Å^3

$Z = 8$

$\mu = 0.35$ mm^{-1}

$T = 293\ (2)$ K

Block, colourless

$0.40 \times 0.10 \times 0.10$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{int} = 0.041$
Radiation source: fine-focus sealed tube	$\theta_{max} = 26.0°$
Monochromator: graphite	$\theta_{min} = 1.9°$
$T = 293(2)$ K	$h = -31 \rightarrow 26$
$\omega/2\theta$ scans	$k = 0 \rightarrow 7$
Absorption correction: ψ scan (North *et al.*, 1968)	$l = 0 \rightarrow 17$
$T_{min} = 0.873,\ T_{max} = 0.966$	3 standard reflections
2002 measured reflections	every 200 reflections
1959 independent reflections	intensity decay: none
1259 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
$R[F^2 > 2\sigma(F^2)] = 0.057$	H-atom parameters constrained
$wR(F^2) = 0.182$	$w = 1/[\sigma^2(F_o{}^2) + (0.1P)^2 + 1.P]$ where $P = (F_o{}^2 + 2F_c{}^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} < 0.001$
1959 reflections	$\Delta\rho_{max} = 0.28$ e Å^{-3}
127 parameters	$\Delta\rho_{min} = -0.30$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. ^{1}H NMR (CDCl$_3$, δ, p.p.m.): 7.12–7.15 (m, 2H), 7.36–7.39 (m, 2H), 4.64 (s, 2H), 2.35 (s, 3H).

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 > 2sigma(F^2) is used only for calculating 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	0.02222 (5)	0.14389 (18)	0.38492 (8)	0.0812 (4)
O	0.08264 (11)	0.5594 (4)	0.5539 (2)	0.0601 (7)
N1	0.13240 (13)	0.7029 (4)	0.5847 (3)	0.0609 (8)
N2	0.16296 (12)	0.3737 (4)	0.5827 (2)	0.0468 (7)
C1	0.41839 (18)	0.8720 (6)	0.7058 (3)	0.0729 (11)
H1B	0.4470	0.7565	0.7324	0.109*
H1C	0.4341	0.9795	0.7603	0.109*
H1D	0.4141	0.9262	0.6407	0.109*
C2	0.35583 (15)	0.7997 (6)	0.6807 (3)	0.0523 (8)
C3	0.34571 (16)	0.5951 (5)	0.6953 (3)	0.0562 (9)
H3A	0.3785	0.5003	0.7223	0.067*
C4	0.28841 (15)	0.5270 (5)	0.6712 (3)	0.0532 (8)
H4A	0.2830	0.3877	0.6823	0.064*
C5	0.23873 (15)	0.6639 (5)	0.6305 (2)	0.0449 (7)
C6	0.24793 (17)	0.8724 (5)	0.6149 (3)	0.0528 (8)
H6A	0.2152	0.9675	0.5877	0.063*
C7	0.30605 (18)	0.9360 (5)	0.6403 (3)	0.0572 (9)
H7A	0.3119	1.0752	0.6299	0.069*
C8	0.17730 (14)	0.5851 (5)	0.5989 (2)	0.0445 (7)
C9	0.10588 (16)	0.3704 (5)	0.5555 (2)	0.0470 (8)
C10	0.06392 (17)	0.1880 (5)	0.5267 (3)	0.0561 (9)
H10A	0.0348	0.2134	0.5489	0.067*
H10B	0.0884	0.0652	0.5650	0.067*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl	0.0833 (8)	0.0822 (8)	0.0651 (6)	−0.0317 (6)	0.0317 (5)	−0.0112 (5)
O	0.0480 (13)	0.0442 (13)	0.0893 (18)	0.0034 (11)	0.0381 (13)	−0.0040 (12)
N1	0.0509 (17)	0.0406 (15)	0.090 (2)	−0.0009 (13)	0.0376 (16)	−0.0041 (14)
N2	0.0492 (16)	0.0377 (14)	0.0543 (15)	−0.0016 (12)	0.0286 (13)	−0.0022 (11)
C1	0.061 (2)	0.083 (3)	0.077 (3)	−0.018 (2)	0.039 (2)	−0.009 (2)
C2	0.052 (2)	0.056 (2)	0.0490 (18)	−0.0086 (16)	0.0272 (16)	−0.0069 (15)
C3	0.0445 (18)	0.057 (2)	0.060 (2)	0.0084 (16)	0.0232 (16)	0.0049 (16)
C4	0.0466 (19)	0.0419 (18)	0.064 (2)	0.0008 (15)	0.0258 (16)	0.0029 (15)
C5	0.0508 (18)	0.0376 (16)	0.0449 (16)	0.0003 (14)	0.0250 (14)	0.0000 (13)
C6	0.059 (2)	0.0407 (18)	0.060 (2)	0.0054 (15)	0.0329 (17)	−0.0006 (15)
C7	0.069 (2)	0.0417 (18)	0.071 (2)	−0.0102 (17)	0.0441 (19)	−0.0065 (16)
C8	0.0460 (18)	0.0393 (17)	0.0467 (17)	0.0028 (14)	0.0241 (14)	0.0009 (12)
C9	0.0516 (19)	0.0436 (18)	0.0482 (17)	0.0025 (14)	0.0287 (15)	−0.0019 (14)
C10	0.056 (2)	0.0485 (19)	0.068 (2)	−0.0053 (16)	0.0358 (17)	0.0027 (16)

Geometric parameters (Å, °)

Cl—C10	1.761 (4)	C3—C4	1.373 (5)

O—C9	1.342 (4)	C3—H3A	0.9300
O—N1	1.426 (4)	C4—C5	1.381 (4)
N1—C8	1.289 (4)	C4—H4A	0.9300
N2—C9	1.282 (4)	C5—C6	1.395 (4)
N2—C8	1.389 (4)	C5—C8	1.460 (5)
C1—C2	1.501 (5)	C6—C7	1.378 (5)
C1—H1B	0.9600	C6—H6A	0.9300
C1—H1C	0.9600	C7—H7A	0.9300
C1—H1D	0.9600	C9—C10	1.482 (4)
C2—C3	1.374 (5)	C10—H10A	0.9700
C2—C7	1.380 (5)	C10—H10B	0.9700
C9—O—N1	105.5 (2)	C6—C5—C8	121.7 (3)
C8—N1—O	103.5 (3)	C7—C6—C5	119.3 (3)
C9—N2—C8	102.6 (3)	C7—C6—H6A	120.3
C2—C1—H1B	109.5	C5—C6—H6A	120.3
C2—C1—H1C	109.5	C6—C7—C2	122.1 (3)
H1B—C1—H1C	109.5	C6—C7—H7A	118.9
C2—C1—H1D	109.5	C2—C7—H7A	118.9
H1B—C1—H1D	109.5	N1—C8—N2	114.5 (3)
H1C—C1—H1D	109.5	N1—C8—C5	123.6 (3)
C3—C2—C7	117.6 (3)	N2—C8—C5	121.9 (3)
C3—C2—C1	121.3 (3)	N2—C9—O	113.9 (3)
C7—C2—C1	121.1 (3)	N2—C9—C10	128.5 (3)
C4—C3—C2	121.7 (3)	O—C9—C10	117.6 (3)
C4—C3—H3A	119.1	C9—C10—Cl	110.0 (2)
C2—C3—H3A	119.1	C9—C10—H10A	109.7
C3—C4—C5	120.4 (3)	Cl—C10—H10A	109.7
C3—C4—H4A	119.8	C9—C10—H10B	109.7
C5—C4—H4A	119.8	Cl—C10—H10B	109.7
C4—C5—C6	118.8 (3)	H10A—C10—H10B	108.2
C4—C5—C8	119.4 (3)		
C9—O—N1—C8	0.8 (3)	C9—N2—C8—N1	1.2 (4)
C7—C2—C3—C4	−0.1 (5)	C9—N2—C8—C5	−179.3 (3)
C1—C2—C3—C4	−179.1 (3)	C4—C5—C8—N1	163.2 (3)
C2—C3—C4—C5	0.3 (5)	C6—C5—C8—N1	−20.5 (5)
C3—C4—C5—C6	−0.3 (5)	C4—C5—C8—N2	−16.2 (5)
C3—C4—C5—C8	176.1 (3)	C6—C5—C8—N2	160.1 (3)
C4—C5—C6—C7	0.1 (5)	C8—N2—C9—O	−0.7 (4)
C8—C5—C6—C7	−176.3 (3)	C8—N2—C9—C10	179.5 (3)
C5—C6—C7—C2	0.1 (5)	N1—O—C9—N2	−0.1 (4)
C3—C2—C7—C6	−0.1 (5)	N1—O—C9—C10	179.8 (3)
C1—C2—C7—C6	178.9 (3)	N2—C9—C10—Cl	−91.1 (4)
O—N1—C8—N2	−1.3 (4)	O—C9—C10—Cl	89.1 (3)
O—N1—C8—C5	179.3 (3)		

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4A···N2	0.93	2.58	2.892 (5)	100

C10—H10B···N1[i]	0.97	2.52	3.436 (5)	157

Symmetry codes: (i) $x, y-1, z$.

Fig. 1