organic compounds

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

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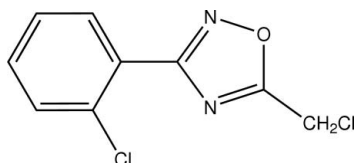
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In the title compound, $C_9H_6Cl_2N_2O$, the dihedral angle between the oxadiazole and benzene ring planes is 12.40 (19)°. Intra- and intermolecular C—H···N interactions occur.

Related literature

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

Experimental

Crystal data

$C_9H_6Cl_2N_2O$	a = 10.396 (2) Å
M_r = 229.06	b = 10.302 (2) Å
Monoclinic, $P2_1/c$	c = 9.2070 (18) Å
β = 98.30 (3)°	μ = 0.63 mm^{-1}
V = 975.7 (3) Å^3	T = 293 (2) K
Z = 4	$0.40 \times 0.20 \times 0.20$ mm
Mo $K\alpha$ radiation	

Data collection

Enraf–Nonius CAD-4 diffractometer	1907 independent reflections
Absorption correction: ψ scan (North *et al.*, 1968)	1204 reflections with $I > 2\sigma(I)$
T_{min} = 0.787, T_{max} = 0.885	R_{int} = 0.037
2033 measured reflections	3 standard reflections
	every 200 reflections
	intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055	127 parameters
$wR(F^2)$ = 0.147	H-atom parameters constrained
S = 1.02	$\Delta\rho_{max}$ = 0.25 e Å^{-3}
1907 reflections	$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1B···N1^i	0.97	2.53	3.317 (5)	138
C9—H9A···N2	0.93	2.44	2.805 (4)	104

Symmetry code: (i) $-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$.

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: HB2637).

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.

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 02 060 439.

supplementary materials

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

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Comment

1,2,4-Oxadiazole derivatives possess biological properties such as intrinsic analgesic (Terashita *et al.*, 2002) and antipicor-naviral (Romero, 2001) effects. As part of out studies in this area, we report here the synthesis and crystal structure of the title compound, (I), (Fig. 1).

The dihedral angles between the N1/C3/N2/C2/O ring and its adjacent bezene ring is 12.40 (19)°.

An intramolecular C—H⋯N hydrogen bond (Table 2) helps to establish the molecular conformation of (I). A short C—H⋯N intermolecular contact is also present.

Experimental

2-Chloro-*N*'-hydroxybenzamidine (24 mmol) was dissolved in 50 ml toluene and the mixture was cooled in an ice bath to at 278 K. 2-Chloroacetyl chloride (30 mmol) in 5 ml toluene was added dropwise. The cold bath was removed, the mixture was refluxed for 1 hr then poured into 50 ml water. Saturated brine (50 ml) was added, and the organic fraction was dried over anhydrous magnesium sulfate and evaporated to dryness to give the title compound (5 g). Colourles blocks of (I) were obtained by slow evaporation of an ethanol solution.

Refinement

All the H atoms were placed geometrically (C—H = 0.93–0.96 Å) and refined as riding with $U_{iso}(H) = 1.2U_{eq}$(carrier) or $1.5U_{eq}$(methyl carrier).
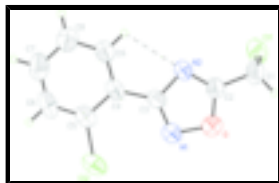
Figures

Fig. 1. A view of the molecular structure of (I) showing 30% displacement ellipsoids (arbitrary spheres for the H atoms). A dashed line indicates the hydrogen bond.

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

Crystal data

$C_9H_6Cl_2N_2O$	$F_{000} = 464$
$M_r = 229.06$	$D_x = 1.559$ Mg m^{-3}

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 10.396$ (2) Å

$b = 10.302$ (2) Å

$c = 9.2070$ (18) Å

$\beta = 98.30$ (3)°

$V = 975.7$ (3) Å^3

$Z = 4$

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Cell parameters from 25 reflections

$\theta = 10–13°$

$\mu = 0.63$ mm^{-1}

$T = 293$ (2) K

Block, colorless

$0.40 \times 0.20 \times 0.20$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293(2)$ K

$\omega/2\theta$ scans

Absorption correction: ψ scan (North *et al.*, 1968)

$T_{min} = 0.787$, $T_{max} = 0.885$

2033 measured reflections

1907 independent reflections

1204 reflections with $I > 2\sigma(I)$

$R_{int} = 0.037$

$\theta_{max} = 26.0°$

$\theta_{min} = 2.0°$

$h = -12 \rightarrow 12$

$k = 0 \rightarrow 12$

$l = 0 \rightarrow 11$

3 standard reflections

every 200 reflections

intensity decay: none

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.055$

$wR(F^2) = 0.147$

$S = 1.02$

1907 reflections

127 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.070P)^2 + 0.3P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.25$ e Å^{-3}

$\Delta\rho_{min} = -0.23$ e Å^{-3}

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*	$U_{iso}*/U_{eq}$
O	0.4532 (2)	0.4767 (2)	0.3282 (3)	0.0654 (7)
Cl1	0.26187 (11)	0.75669 (10)	0.16907 (14)	0.0813 (4)
N1	0.4105 (3)	0.3594 (3)	0.3838 (3)	0.0632 (8)
C1	0.3883 (4)	0.7009 (3)	0.3021 (4)	0.0642 (10)
H1B	0.4699	0.7047	0.2628	0.077*
H1C	0.3954	0.7568	0.3876	0.077*
Cl2	0.34090 (11)	0.08541 (9)	0.42588 (14)	0.0843 (4)
N2	0.2688 (2)	0.5213 (2)	0.4048 (3)	0.0481 (7)
C2	0.3640 (3)	0.5652 (3)	0.3462 (4)	0.0494 (8)
C3	0.3010 (3)	0.3917 (3)	0.4273 (3)	0.0428 (7)
C4	0.2168 (3)	0.3062 (3)	0.4985 (3)	0.0465 (8)
C5	0.2250 (3)	0.1701 (3)	0.5042 (4)	0.0537 (9)
C6	0.1378 (4)	0.0992 (3)	0.5721 (4)	0.0667 (11)
H6A	0.1437	0.0091	0.5747	0.080*
C7	0.0428 (4)	0.1597 (4)	0.6353 (4)	0.0715 (11)
H7A	−0.0151	0.1109	0.6809	0.086*
C8	0.0330 (4)	0.2935 (4)	0.6316 (4)	0.0678 (10)
H8A	−0.0314	0.3351	0.6744	0.081*
C9	0.1189 (3)	0.3641 (4)	0.5643 (4)	0.0571 (9)
H9A	0.1116	0.4541	0.5625	0.069*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O	0.0661 (16)	0.0520 (14)	0.0853 (19)	0.0014 (12)	0.0355 (13)	0.0024 (13)
Cl1	0.0925 (8)	0.0579 (6)	0.0945 (8)	0.0035 (5)	0.0167 (6)	0.0286 (5)
N1	0.0640 (18)	0.0398 (15)	0.091 (2)	0.0035 (14)	0.0301 (16)	0.0038 (16)
C1	0.073 (2)	0.053 (2)	0.069 (2)	−0.0124 (18)	0.0208 (19)	0.0089 (18)
Cl2	0.0969 (8)	0.0412 (5)	0.1189 (10)	0.0123 (5)	0.0296 (7)	−0.0026 (5)
N2	0.0528 (16)	0.0344 (14)	0.0580 (17)	−0.0009 (12)	0.0110 (12)	0.0010 (12)
C2	0.0544 (19)	0.0433 (17)	0.052 (2)	−0.0036 (15)	0.0120 (15)	0.0020 (15)
C3	0.0495 (17)	0.0357 (16)	0.0430 (17)	0.0035 (13)	0.0061 (14)	0.0001 (13)
C4	0.0553 (19)	0.0365 (16)	0.0456 (19)	−0.0013 (14)	0.0004 (15)	0.0046 (14)
C5	0.061 (2)	0.0424 (18)	0.055 (2)	−0.0002 (16)	−0.0026 (16)	0.0017 (16)
C6	0.077 (3)	0.045 (2)	0.072 (3)	−0.0131 (19)	−0.008 (2)	0.0180 (18)
C7	0.064 (2)	0.072 (3)	0.078 (3)	−0.016 (2)	0.009 (2)	0.021 (2)
C8	0.067 (2)	0.066 (2)	0.073 (3)	−0.004 (2)	0.019 (2)	0.008 (2)
C9	0.058 (2)	0.0467 (18)	0.069 (2)	0.0019 (16)	0.0153 (17)	0.0087 (17)

Geometric parameters (Å, °)

O—C2	1.328 (4)	C4—C9	1.392 (4)

O—N1	1.409 (3)	C4—C5	1.406 (4)
Cl1—C1	1.758 (4)	C5—C6	1.382 (5)
N1—C3	1.303 (4)	C6—C7	1.367 (5)
C1—C2	1.487 (4)	C6—H6A	0.9300
C1—H1B	0.9700	C7—C8	1.382 (6)
C1—H1C	0.9700	C7—H7A	0.9300
Cl2—C5	1.727 (4)	C8—C9	1.367 (5)
N2—C2	1.276 (4)	C8—H8A	0.9300
N2—C3	1.385 (4)	C9—H9A	0.9300
C3—C4	1.462 (4)		
C2—O—N1	106.3 (2)	C5—C4—C3	125.5 (3)
C3—N1—O	103.2 (2)	C6—C5—C4	120.2 (3)
C2—C1—Cl1	111.0 (3)	C6—C5—Cl2	117.7 (3)
C2—C1—H1B	109.4	C4—C5—Cl2	122.1 (3)
Cl1—C1—H1B	109.4	C7—C6—C5	120.9 (3)
C2—C1—H1C	109.4	C7—C6—H6A	119.6
Cl1—C1—H1C	109.4	C5—C6—H6A	119.6
H1B—C1—H1C	108.0	C6—C7—C8	120.0 (4)
C2—N2—C3	102.7 (3)	C6—C7—H7A	120.0
N2—C2—O	114.1 (3)	C8—C7—H7A	120.0
N2—C2—C1	128.4 (3)	C9—C8—C7	119.4 (4)
O—C2—C1	117.5 (3)	C9—C8—H8A	120.3
N1—C3—N2	113.8 (3)	C7—C8—H8A	120.3
N1—C3—C4	126.2 (3)	C8—C9—C4	122.4 (3)
N2—C3—C4	120.0 (3)	C8—C9—H9A	118.8
C9—C4—C5	117.1 (3)	C4—C9—H9A	118.8
C9—C4—C3	117.4 (3)		
C2—O—N1—C3	0.4 (4)	N1—C3—C4—C5	−14.3 (5)
C3—N2—C2—O	0.4 (4)	N2—C3—C4—C5	167.8 (3)
C3—N2—C2—C1	−177.2 (3)	C9—C4—C5—C6	0.4 (5)
N1—O—C2—N2	−0.5 (4)	C3—C4—C5—C6	−178.5 (3)
N1—O—C2—C1	177.4 (3)	C9—C4—C5—Cl2	179.5 (3)
Cl1—C1—C2—N2	−60.6 (5)	C3—C4—C5—Cl2	0.6 (5)
Cl1—C1—C2—O	121.9 (3)	C4—C5—C6—C7	−0.4 (5)
O—N1—C3—N2	−0.1 (4)	Cl2—C5—C6—C7	−179.5 (3)
O—N1—C3—C4	−178.1 (3)	C5—C6—C7—C8	0.2 (6)
C2—N2—C3—N1	−0.1 (4)	C6—C7—C8—C9	0.0 (6)
C2—N2—C3—C4	178.0 (3)	C7—C8—C9—C4	0.1 (6)
N1—C3—C4—C9	166.8 (3)	C5—C4—C9—C8	−0.3 (5)
N2—C3—C4—C9	−11.1 (4)	C3—C4—C9—C8	178.7 (3)

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1B···N1[i]	0.97	2.53	3.317 (5)	138
C9—H9A···N2	0.93	2.44	2.805 (4)	104

Symmetry codes: (i) −x+1, y+1/2, −z+1/2.

Fig. 1