Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Diclomezine: 6-(3,5-dichloro-4-methylphenyl)pyridazin-3(2*H*)-one

Hyunjee Kim, Tae Ho Kim,* Jineun Kim and Ki-Min Park*

Department of Chemistry and Research Institute of Natural Sciences, Gyeongsang National University, Jinju 660-701, Republic of Korea
Correspondence e-mail: thkim@gnu.ac.kr, kmpark@gnu.ac.kr

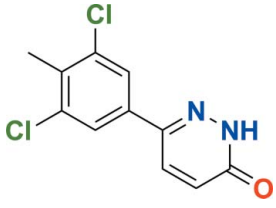
Received 12 November 2010; accepted 16 November 2010

Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; R factor = 0.034; wR factor = 0.091; data-to-parameter ratio = 18.2.

In the title compound, $C_{11}H_8Cl_2N_2O$, the benzene and pyridazine rings are tilted by 8.6 (1)° relative to each other. In the crystal, pairs of intermolecular N—H···O hydrogen bonds form centrosymmetric dimers. π–π contacts with centroid–centroid distances of 3.698 (2) and 3.751 (1) Å and halogen–halogen interactions [3.379 (1) Å] also stabilize the structure.

Related literature

For information on the toxicity and fungicidal properties of the title compound, see: Sankyo (1998). For a related structure, see: Prout *et al.* (1994).



Experimental

Crystal data

$C_{11}H_8Cl_2N_2O$
M_r = 255.09
Monoclinic, $P2_1/c$
a = 9.745 (4) Å
b = 13.850 (5) Å
c = 8.481 (3) Å
β = 111.557 (6)°
V = 1064.7 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.59 mm^{-1}
T = 173 K
0.19 × 0.09 × 0.08 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.897, T_{max} = 0.955
10410 measured reflections
2657 independent reflections
2038 reflections with $I > 2\sigma(I)$
R_{int} = 0.042

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.091
S = 1.07
2657 reflections
146 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.88	1.90	2.771 (2)	172

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

Data collection: *APEX2* (Bruker, 2006); cell refinement: *SAINT* (Bruker, 2006); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* and *DIAMOND* (Brandenburg, 1998).

This research was supported by the Basic Science Research Program through the National Research Foundation of Korea (NRF) funded by the Ministry of Education, Science and Technology (No. 2010–0009089).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ5054).

References

Brandenburg, K. (1998). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Bruker (2006). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Prout, K., Bannister, C., Burns, K., Chen, M., Warrington, B. H. & Vinter, J. G. (1994). *Acta Cryst.* B**50**, 71–85.
Sankyo (1998). *J. Pestic. Sci.*, **13**, 625–628
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2010). E**66**, o3257 [https://doi.org/10.1107/S1600536810047409]

Diclomezine: 6-(3,5-dichloro-4-methylphenyl)pyridazin-3(2*H*)-one

Hyunjee Kim, Tae Ho Kim, Jineun Kim and Ki-Min Park

S1. Comment

Declomezine (systematic name: 6-(3,5-dichloro-4-methylphenyl)- 3(*2H*)-pyridazinone), is a well known fungicide (Sankyo 1998). In this paper, the structure of the title pyridazinone derivative is reported.

In the molecular structure of the title compound (Scheme 1, Fig. 1), the dihedral angle between the phenyl ring and the pyridazine ring is 8.6 (1) °, compared to the value of 18.0 ° in the similar compound 6-phenyl-3(*2H*)-pyridazinone (Prout *et al.*, 1994). Bond lengths and angles observed here are also similar to those in 6-phenyl-3(*2H*)-pyridazinone.

In the crystal structure, as shown in Fig. 2, there are pair-wise intermolecular N—H···O hydrogen bonds (Table 1; symmetry code as in Fig. 2). Weak intermolecular π—π and halogen···halogen interactions also exist [$Cg1\cdots Cg2^{iv}$ 3.75 Å, $Cg2\cdots Cg2^{v}$ 3.70 Å and $Cl1\cdots Cl2^{iii}$ 3.379 (1) Å; *Cg*1 and *Cg*2 are the centroids of the phenyl and pyridazine rings, respectively.]. These intermolecular interactions contribute to the stabilization of the packing.

S2. Experimental

The title compound was purchased from the Dr. Ehrenstorfer GmbH Company. Slow evaporation of a solution in CH_2Cl_2 gave single crystals suitable for X-ray analysis.

S3. Refinement

All H-atoms were positioned geometrically and refined using a riding model with d(N—H) = 0.88 Å, $U_{iso} = 1.2U_{eq}$(N) for pyrazine, d(C—H) = 0.95 Å, $U_{iso} = 1.2U_{eq}$(C) for aromatic, d(C—H) = 0.98 Å, $U_{iso} = 1.5U_{eq}$(C) for methyl protons.

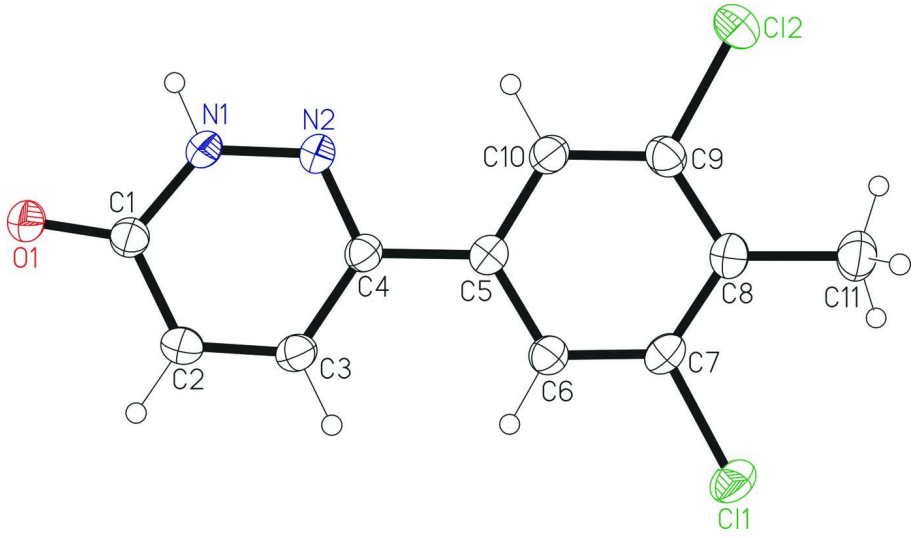


Figure 1

The molecular structure of the title compound, showing displacement ellipsoids drawn at the 50% probability level.

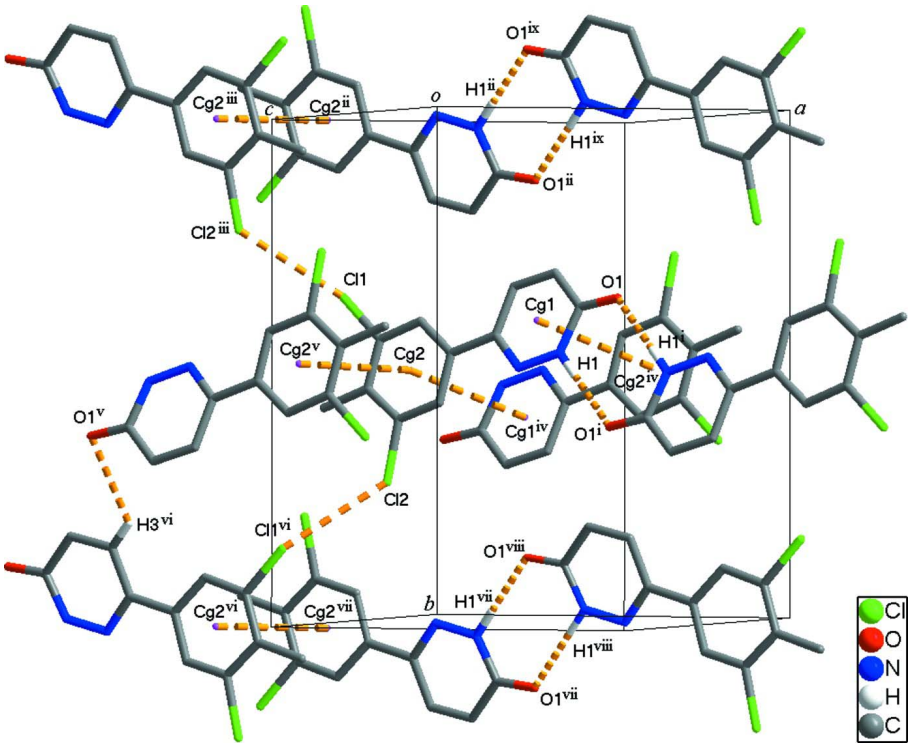


Figure 2

Crystal packing of the title compound with intermolecular N—H···O hydrogen bonds, π—π and halogen—halogen interactions shown as dashed lines. H atoms not involved in intermolecular interactions have been omitted for clarity. *Cg*1 and *Cg*2 are the centroids of the phenyl and pyridazine rings, respectively. (Symmetry codes: i) 1 - *x*, 1 - *y*, -*z*; ii) *x*, 0.5 - *y*, 1/2 + *z*; iii) -*x*, -1/2 + *y*, 1.5 - *z*; iv) 1 - *x*, 1 - *y*, 1 - *z*; v) -*x*, 1 - *y*, 1 - *z*; vi) -*x*, 1/2 + *y*, 1.5 - *z*; vii) *x*, 1.5 - *y*, 1/2 + *z*; viii) 1 - *x*, 1/2 + *y*, 0.5 - *z*; ix) 1 - *x*, -1/2 + *y*, 0.5 - *z*) .

6-(3,5-dichloro-4-methylphenyl)pyridazin-3(2*H*)-one

Crystal data

$C_{11}H_8Cl_2N_2O$
M_r = 255.09
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 9.745 (4) Å
b = 13.850 (5) Å
c = 8.481 (3) Å
β = 111.557 (6)°
V = 1064.7 (7) Å^3
Z = 4
F(000) = 520
D_x = 1.591 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3791 reflections
θ = 2.9–28.2°
μ = 0.59 mm^{-1}
T = 173 K
Block, colourless
0.19 × 0.09 × 0.08 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans

Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.897, T_{max} = 0.955
10410 measured reflections
2657 independent reflections
2038 reflections with $I > 2\sigma(I)$

R_{int} = 0.042
θ_{max} = 28.4°, θ_{min} = 2.3°
$h = -12 \rightarrow 13$
$k = -18 \rightarrow 17$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.091
S = 1.07
2657 reflections
146 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0323P)^2 + 0.5573P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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}
Cl1	0.11909 (5)	0.35378 (4)	0.82090 (6)	0.03367 (14)
Cl2	0.15267 (6)	0.72198 (3)	0.62638 (7)	0.03884 (15)
O1	0.52503 (15)	0.37232 (9)	0.02888 (17)	0.0290 (3)
N1	0.42007 (16)	0.47759 (11)	0.15589 (18)	0.0240 (3)
H1	0.4313	0.5229	0.0888	0.029*
N2	0.35554 (16)	0.50487 (11)	0.26436 (18)	0.0239 (3)
C1	0.47026 (19)	0.38769 (13)	0.1379 (2)	0.0234 (4)
C2	0.45389 (19)	0.31751 (13)	0.2541 (2)	0.0258 (4)
H2	0.4893	0.2536	0.2543	0.031*
C3	0.38829 (19)	0.34224 (13)	0.3631 (2)	0.0246 (4)
H3	0.3760	0.2954	0.4387	0.029*
C4	0.33735 (18)	0.43848 (12)	0.3652 (2)	0.0214 (3)
C5	0.26737 (18)	0.47099 (12)	0.4848 (2)	0.0218 (3)
C6	0.22820 (18)	0.40586 (13)	0.5865 (2)	0.0239 (4)
H6	0.2436	0.3386	0.5781	0.029*
C7	0.16682 (18)	0.43931 (13)	0.6996 (2)	0.0243 (4)
C8	0.13991 (18)	0.53629 (13)	0.7192 (2)	0.0241 (4)
C9	0.17893 (19)	0.59892 (13)	0.6130 (2)	0.0254 (4)
C10	0.23982 (18)	0.56857 (13)	0.4983 (2)	0.0238 (4)

H10	0.2630	0.6143	0.4284	0.029*
C11	0.0760 (2)	0.57125 (15)	0.8451 (2)	0.0332 (4)
H11C	0.1557	0.5816	0.9551	0.050*
H11A	0.0072	0.5229	0.8573	0.050*
H11B	0.0235	0.6321	0.8053	0.050*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0373 (3)	0.0338 (3)	0.0379 (3)	−0.00164 (19)	0.0233 (2)	0.0084 (2)
Cl2	0.0529 (3)	0.0248 (2)	0.0479 (3)	0.0092 (2)	0.0292 (3)	0.0019 (2)
O1	0.0396 (7)	0.0248 (7)	0.0304 (7)	0.0001 (5)	0.0220 (6)	−0.0012 (5)
N1	0.0319 (8)	0.0224 (7)	0.0227 (8)	0.0001 (6)	0.0157 (6)	0.0023 (6)
N2	0.0283 (7)	0.0235 (7)	0.0240 (8)	0.0003 (6)	0.0147 (6)	0.0006 (6)
C1	0.0247 (8)	0.0223 (8)	0.0242 (9)	−0.0025 (6)	0.0101 (7)	−0.0022 (7)
C2	0.0304 (9)	0.0192 (8)	0.0292 (10)	−0.0006 (7)	0.0127 (8)	0.0000 (7)
C3	0.0286 (9)	0.0219 (9)	0.0250 (9)	−0.0022 (7)	0.0120 (7)	0.0017 (7)
C4	0.0222 (8)	0.0216 (8)	0.0210 (8)	−0.0020 (6)	0.0087 (7)	−0.0003 (7)
C5	0.0202 (8)	0.0243 (9)	0.0203 (8)	−0.0014 (6)	0.0068 (6)	0.0003 (7)
C6	0.0239 (8)	0.0230 (9)	0.0265 (9)	−0.0003 (7)	0.0112 (7)	0.0009 (7)
C7	0.0226 (8)	0.0285 (9)	0.0231 (9)	−0.0019 (7)	0.0099 (7)	0.0037 (7)
C8	0.0197 (8)	0.0303 (9)	0.0220 (9)	0.0008 (7)	0.0072 (7)	−0.0012 (7)
C9	0.0261 (9)	0.0233 (9)	0.0266 (9)	0.0038 (7)	0.0096 (7)	0.0002 (7)
C10	0.0241 (8)	0.0244 (9)	0.0246 (9)	0.0010 (7)	0.0109 (7)	0.0037 (7)
C11	0.0374 (10)	0.0373 (11)	0.0296 (10)	0.0043 (8)	0.0178 (8)	−0.0001 (9)

Geometric parameters (Å, º)

Cl1—C7	1.7404 (18)	C5—C10	1.391 (3)
Cl2—C9	1.733 (2)	C5—C6	1.395 (2)
O1—C1	1.244 (2)	C6—C7	1.384 (2)
N1—N2	1.345 (2)	C6—H6	0.9500
N1—C1	1.367 (2)	C7—C8	1.390 (3)
N1—H1	0.8800	C8—C9	1.400 (3)
N2—C4	1.311 (2)	C8—C11	1.500 (2)
C1—C2	1.435 (3)	C9—C10	1.378 (2)
C2—C3	1.347 (3)	C10—H10	0.9500
C2—H2	0.9500	C11—H11C	0.9800
C3—C4	1.425 (2)	C11—H11A	0.9800
C3—H3	0.9500	C11—H11B	0.9800
C4—C5	1.485 (2)		
N2—N1—C1	127.50 (15)	C7—C6—H6	120.1
N2—N1—H1	116.2	C5—C6—H6	120.1
C1—N1—H1	116.2	C6—C7—C8	123.71 (16)
C4—N2—N1	117.27 (15)	C6—C7—Cl1	117.35 (14)
O1—C1—N1	120.54 (16)	C8—C7—Cl1	118.93 (14)
O1—C1—C2	125.54 (17)	C7—C8—C9	114.45 (16)

N1—C1—C2	113.92 (16)	C7—C8—C11	122.88 (17)
C3—C2—C1	120.00 (17)	C9—C8—C11	122.67 (17)
C3—C2—H2	120.0	C10—C9—C8	123.67 (17)
C1—C2—H2	120.0	C10—C9—Cl2	117.27 (14)
C2—C3—C4	120.11 (16)	C8—C9—Cl2	119.06 (14)
C2—C3—H3	119.9	C9—C10—C5	120.05 (16)
C4—C3—H3	119.9	C9—C10—H10	120.0
N2—C4—C3	121.12 (16)	C5—C10—H10	120.0
N2—C4—C5	116.03 (15)	C8—C11—H11C	109.5
C3—C4—C5	122.79 (15)	C8—C11—H11A	109.5
C10—C5—C6	118.21 (16)	H11C—C11—H11A	109.5
C10—C5—C4	120.12 (15)	C8—C11—H11B	109.5
C6—C5—C4	121.67 (16)	H11C—C11—H11B	109.5
C7—C6—C5	119.90 (17)	H11A—C11—H11B	109.5

C1—N1—N2—C4	−0.2 (3)	C4—C5—C6—C7	178.27 (15)
N2—N1—C1—O1	178.37 (16)	C5—C6—C7—C8	0.3 (3)
N2—N1—C1—C2	−2.0 (3)	C5—C6—C7—Cl1	179.41 (13)
O1—C1—C2—C3	−177.86 (17)	C6—C7—C8—C9	0.6 (3)
N1—C1—C2—C3	2.6 (2)	Cl1—C7—C8—C9	−178.51 (13)
C1—C2—C3—C4	−1.1 (3)	C6—C7—C8—C11	−178.75 (17)
N1—N2—C4—C3	1.9 (2)	Cl1—C7—C8—C11	2.2 (2)
N1—N2—C4—C5	179.33 (14)	C7—C8—C9—C10	−0.3 (3)
C2—C3—C4—N2	−1.3 (3)	C11—C8—C9—C10	178.98 (17)
C2—C3—C4—C5	−178.51 (16)	C7—C8—C9—Cl2	−179.37 (13)
N2—C4—C5—C10	−6.9 (2)	C11—C8—C9—Cl2	−0.1 (2)
C3—C4—C5—C10	170.44 (16)	C8—C9—C10—C5	−0.8 (3)
N2—C4—C5—C6	173.38 (16)	Cl2—C9—C10—C5	178.26 (13)
C3—C4—C5—C6	−9.3 (3)	C6—C5—C10—C9	1.7 (3)
C10—C5—C6—C7	−1.5 (3)	C4—C5—C10—C9	−178.06 (15)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.88	1.90	2.771 (2)	172

Symmetry code: (i) −*x*+1, −*y*+1, −*z*.