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N^1,N^3-Di-2-pyridylmalonamide

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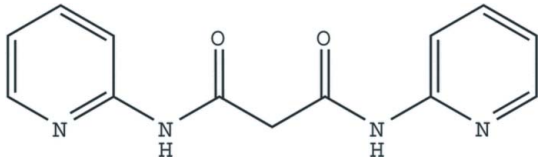
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.040; wR factor = 0.105; data-to-parameter ratio = 14.8.

The title compound, $C_{13}H_{12}N_4O_2$, consists of two pyridine rings connected by a malonamide chain. The dihedral angle between the two pyridine rings is 88.84 (7)°. The crystal packing is stabilized by N—H···O hydrogen bonds.

Related literature

For related literature, see: Chu *et al.* (2007); Shoukry *et al.* (2004); Yolanda *et al.* (2002); Siddall (1960); Gasparini & Grossi (1980); Goujon & Shipman (2002).



Experimental

Crystal data

$C_{13}H_{12}N_4O_2$
M_r = 256.27
Monoclinic, $P2_1/c$
a = 13.112 (9) Å
b = 9.412 (5) Å
c = 9.799 (5) Å
β = 94.30 (3)°
V = 1205.9 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 298 (2) K
0.60 × 0.40 × 0.10 mm

Data collection

Rigaku R-AXIS RAPID imaging plate system diffractometer
Absorption correction: none
11080 measured reflections
2732 independent reflections
1970 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.105
S = 1.02
2732 reflections
184 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H1···O1^{i}	0.86	2.17	3.026 (2)	172
N3—H2···O2^{ii}	0.86	2.08	2.9272 (19)	169

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

Data collection: *RAPID-AUTO* (Rigaku, 2006); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEX* (McArdle, 1995); software used to prepare material for publication: *SHELXL97*-2.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LX2007).

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supplementary materials

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N^1,N^3-Di-2-pyridylmalonamide

H.-Y. Yu, X. Fang, M.-L. Cao and J.-D. Wang

Comment

The title compound, (I), is a derivative of malonamides who were proved to be effective and selective κ optical receptor agonists (Chu *et al.*, 2007). These compounds can coordinate to the alkali metals, alkaline earth metals and transition metals easily (Shoukry *et al.*, 2004,

Yolanda *et al.*, 2002). Simultaneity, they are also the 'green' extraction of quadrivalent actinides and zirconium (Siddall, 1960, Gasparini & Grossi, 1980).

The molecular structure of (I) is shown in Fig. 1. The dihedral angle between two pyridine rings of one molecule is 88.84 (7)°. In (I), the molecules are packed with hydrogen bonds of N—H···O as listed in Table 1 and π-π interactions are not observed between the aromatic pyridine rings.

Experimental

The diethyl malonate (1.69 g 10.6 mmol) was added to a solution of pyridin-2-amine (2.50 g 27 mmol) in xylene (15 ml, 141 mmol) carefully and then the solution was refluxed at 413 K for 8 hrs(Goujon & Shipman, 2002). After cooling to room temperature, the mixture was filtered, the solid crude product was dried at 80 °C and recrystallized from a mixture of toluene and DMF (10 : 1) to give colorless crystals, in yield of 31%.

Figures

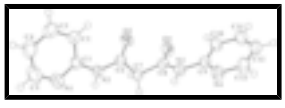

Fig. 1. A view of the molecular structure of (I), with displacement ellipsoids drawn at the 50% probabilty level for non-H atoms.

N^1,N^3—Di-2-pyridylmalonamide

Crystal data

$C_{13}H_{12}N_4O_2$	F_{000} = 536
M_r = 256.27	D_x = 1.412 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71069 Å
Hall symbol: -P2ybc	Cell parameters from 7891 reflections
a = 13.112 (9) Å	θ = 6.0–55.3º
b = 9.412 (5) Å	μ = 0.10 mm^{-1}
c = 9.799 (5) Å	T = 298 (2) K
β = 94.30 (3)º	Block, colorless

V = 1205.9 (12) Å^3	0.60 × 0.40 × 0.10 mm
Z = 4	

Data collection

Rigaku model???? imaging plate system diffractometer	2732 independent reflections
Radiation source: Rigaku rotating anode generator	1970 reflections with $I > 2\sigma(I)$
Monochromator: Graphite Monochromator	R_{int} = 0.033
Detector resolution: 10 pixels mm^{-1}	θ_{max} = 27.5º
T = 298(2) K	θ_{min} = 3.0º
imaging plate scans	h = −17→16
Absorption correction: none	k = −12→12
11080 measured reflections	l = −11→12

Refinement

Refinement on F^2	Only H-atom displacement parameters refined
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0497P)^2 + 0.1727P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.040	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.105	$\Delta\rho_{max}$ = 0.15 e Å^{-3}
S = 1.02	$\Delta\rho_{min}$ = −0.19 e Å^{-3}
2732 reflections	Extinction correction: none
184 parameters	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	
Hydrogen site location: inferred from neighbouring sites	

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 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}
N1	0.60032 (11)	0.40681 (15)	0.35915 (12)	0.0532 (4)
N2	0.74584 (9)	0.28807 (13)	0.31452 (11)	0.0404 (3)

H1	0.7615	0.2972	0.4009	0.064 (5)*
N3	1.04889 (9)	0.27078 (11)	0.17253 (12)	0.0373 (3)
H2	1.0310	0.3543	0.1972	0.053 (5)*
N4	1.15370 (10)	0.39584 (13)	0.04360 (14)	0.0498 (3)
O1	0.80805 (8)	0.20945 (12)	0.11820 (9)	0.0506 (3)
O2	1.01988 (9)	0.03660 (10)	0.20936 (11)	0.0504 (3)
C1	0.64675 (11)	0.33103 (15)	0.26822 (13)	0.0374 (3)
C2	0.60119 (12)	0.29727 (19)	0.14094 (15)	0.0509 (4)
H3	0.6353	0.2426	0.0797	0.064 (5)*
C3	0.50414 (14)	0.3465 (2)	0.10707 (17)	0.0628 (5)
H4	0.4720	0.3263	0.0214	0.087 (7)*
C4	0.45468 (14)	0.4249 (2)	0.19851 (18)	0.0647 (5)
H5	0.3888	0.4589	0.1773	0.079 (6)*
C5	0.50561 (15)	0.4517 (2)	0.32254 (18)	0.0657 (5)
H6	0.4721	0.5047	0.3856	0.074 (6)*
C6	0.81999 (11)	0.23400 (13)	0.24058 (13)	0.0340 (3)
C7	0.91980 (11)	0.20588 (14)	0.32271 (13)	0.0351 (3)
H7	0.9103	0.1313	0.3889	0.047 (4)*
H8	0.9416	0.2911	0.3724	0.046 (4)*
C8	1.00105 (11)	0.16180 (13)	0.23043 (13)	0.0351 (3)
C9	1.12351 (11)	0.26680 (14)	0.07794 (13)	0.0360 (3)
C10	1.16187 (13)	0.14448 (16)	0.02381 (16)	0.0499 (4)
H9	1.1396	0.0554	0.0500	0.069 (5)*
C11	1.23391 (14)	0.15810 (18)	−0.06999 (18)	0.0564 (4)
H10	1.2614	0.0774	−0.1079	0.075 (6)*
C12	1.26539 (13)	0.28983 (18)	−0.10785 (16)	0.0509 (4)
H11	1.3139	0.3008	−0.1716	0.066 (5)*
C13	1.22311 (13)	0.40432 (18)	−0.04889 (17)	0.0545 (4)
H12	1.2439	0.4942	−0.0747	0.071 (5)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0513 (8)	0.0685 (9)	0.0401 (6)	0.0177 (7)	0.0053 (6)	−0.0027 (6)
N2	0.0355 (7)	0.0557 (7)	0.0300 (5)	0.0019 (5)	0.0025 (5)	−0.0042 (5)
N3	0.0372 (7)	0.0301 (5)	0.0457 (6)	−0.0003 (5)	0.0106 (5)	−0.0011 (5)
N4	0.0505 (8)	0.0397 (6)	0.0617 (8)	−0.0026 (6)	0.0205 (6)	0.0044 (6)
O1	0.0454 (7)	0.0736 (7)	0.0329 (5)	0.0052 (5)	0.0033 (4)	−0.0088 (5)
O2	0.0584 (7)	0.0315 (5)	0.0642 (7)	−0.0004 (4)	0.0233 (5)	0.0000 (5)
C1	0.0351 (8)	0.0425 (7)	0.0353 (6)	−0.0021 (6)	0.0067 (5)	0.0041 (6)
C2	0.0391 (9)	0.0707 (10)	0.0429 (8)	−0.0039 (7)	0.0036 (6)	−0.0112 (8)
C3	0.0421 (10)	0.0954 (14)	0.0496 (9)	−0.0013 (9)	−0.0055 (7)	−0.0036 (9)
C4	0.0448 (10)	0.0911 (13)	0.0574 (10)	0.0175 (9)	−0.0003 (8)	0.0093 (9)
C5	0.0582 (12)	0.0862 (13)	0.0532 (9)	0.0296 (10)	0.0072 (8)	−0.0011 (9)
C6	0.0356 (8)	0.0339 (6)	0.0329 (6)	−0.0047 (5)	0.0055 (5)	0.0012 (5)
C7	0.0363 (8)	0.0352 (7)	0.0339 (6)	−0.0017 (5)	0.0036 (5)	0.0021 (6)
C8	0.0356 (7)	0.0324 (6)	0.0370 (6)	−0.0011 (5)	0.0019 (5)	0.0011 (6)
C9	0.0302 (7)	0.0384 (7)	0.0395 (7)	−0.0014 (5)	0.0024 (5)	0.0016 (6)

C10	0.0503 (10)	0.0404 (8)	0.0611 (9)	0.0011 (7)	0.0188 (7)	−0.0017 (7)
C11	0.0516 (11)	0.0566 (10)	0.0635 (10)	0.0053 (8)	0.0198 (8)	−0.0086 (8)
C12	0.0369 (9)	0.0689 (10)	0.0482 (8)	−0.0027 (7)	0.0120 (7)	0.0027 (8)
C13	0.0508 (10)	0.0520 (9)	0.0628 (10)	−0.0056 (7)	0.0184 (8)	0.0095 (8)

Geometric parameters (Å, °)

N1—C1	1.325 (2)	C3—H4	0.9300
N1—C5	1.335 (2)	C4—C5	1.366 (3)
N2—C6	1.355 (2)	C4—H5	0.9300
N2—C1	1.403 (2)	C5—H6	0.9300
N2—H1	0.8600	C6—C7	1.508 (2)
N3—C8	1.349 (2)	C7—C8	1.506 (2)
N3—C9	1.398 (2)	C7—H7	0.9700
N3—H2	0.8600	C7—H8	0.9700
N4—C9	1.329 (2)	C9—C10	1.378 (2)
N4—C13	1.334 (2)	C10—C11	1.372 (2)
O1—C6	1.220 (2)	C10—H9	0.9300
O2—C8	1.225 (2)	C11—C12	1.367 (2)
C1—C2	1.379 (2)	C11—H10	0.9300
C2—C3	1.372 (3)	C12—C13	1.360 (2)
C2—H3	0.9300	C12—H11	0.9300
C3—C4	1.362 (3)	C13—H12	0.9300
C1—N1—C5	117.06 (14)	N2—C6—C7	114.18 (12)
C6—N2—C1	128.29 (12)	C6—C7—C8	110.65 (11)
C6—N2—H1	115.9	C6—C7—H7	109.5
C1—N2—H1	115.9	C8—C7—H7	109.5
C8—N3—C9	128.98 (11)	C6—C7—H8	109.5
C8—N3—H2	115.5	C8—C7—H8	109.5
C9—N3—H2	115.5	H7—C7—H8	108.1
C9—N4—C13	117.27 (13)	O2—C8—N3	123.68 (13)
N1—C1—C2	122.87 (14)	O2—C8—C7	121.79 (12)
N1—C1—N2	113.50 (12)	N3—C8—C7	114.52 (11)
C2—C1—N2	123.63 (13)	N4—C9—C10	122.81 (14)
C3—C2—C1	118.17 (15)	N4—C9—N3	112.34 (12)
C3—C2—H3	120.9	C10—C9—N3	124.84 (12)
C1—C2—H3	120.9	C11—C10—C9	117.97 (14)
C4—C3—C2	120.18 (16)	C11—C10—H9	121.0
C4—C3—H4	119.9	C9—C10—H9	121.0
C2—C3—H4	119.9	C12—C11—C10	120.23 (15)
C5—C4—C3	117.41 (17)	C12—C11—H10	119.9
C5—C4—H5	121.3	C10—C11—H10	119.9
C3—C4—H5	121.3	C11—C12—C13	117.54 (15)
N1—C5—C4	124.30 (16)	C11—C12—H11	121.2
N1—C5—H6	117.8	C13—C12—H11	121.2
C4—C5—H6	117.8	N4—C13—C12	124.17 (15)
O1—C6—N2	123.55 (13)	N4—C13—H12	117.9
O1—C6—C7	122.27 (13)	C12—C13—H12	117.9
C5—N1—C1—C2	−0.5 (2)	C9—N3—C8—O2	2.3 (2)

C5—N1—C1—N2	179.97 (15)	C9—N3—C8—C7	−176.65 (13)
C6—N2—C1—N1	−166.38 (13)	C6—C7—C8—O2	−95.13 (15)
C6—N2—C1—C2	14.1 (2)	C6—C7—C8—N3	83.80 (14)
N1—C1—C2—C3	1.0 (2)	C13—N4—C9—C10	1.0 (2)
N2—C1—C2—C3	−179.50 (15)	C13—N4—C9—N3	−178.39 (14)
C1—C2—C3—C4	−0.8 (3)	C8—N3—C9—N4	−179.77 (14)
C2—C3—C4—C5	0.2 (3)	C8—N3—C9—C10	0.9 (2)
C1—N1—C5—C4	−0.2 (3)	N4—C9—C10—C11	−0.3 (2)
C3—C4—C5—N1	0.4 (3)	N3—C9—C10—C11	178.94 (15)
C1—N2—C6—O1	−3.0 (2)	C9—C10—C11—C12	−0.3 (3)
C1—N2—C6—C7	177.10 (12)	C10—C11—C12—C13	0.3 (3)
O1—C6—C7—C8	6.48 (18)	C9—N4—C13—C12	−1.0 (3)
N2—C6—C7—C8	−173.63 (11)	C11—C12—C13—N4	0.4 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H1···O1^{i}	0.86	2.17	3.026 (2)	172
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Symmetry codes: (i) *x*, −*y*+1/2, *z*+1/2; (ii) −*x*+2, *y*+1/2, −*z*+1/2.

Fig. 1