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Bis(3,5-dimethylpyrazol-1-yl)acetic acid

Quang Trung Nguyen and Jong Hwa Jeong*

Department of Chemistry, Kyungpook National University, Taegu 702-701, Republic of Korea
Correspondence e-mail: jeongjh@knu.ac.kr

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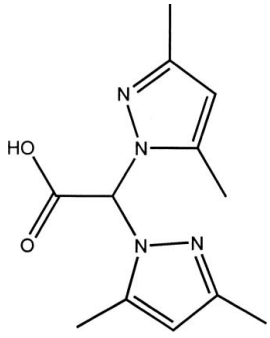
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.040; wR factor = 0.120; data-to-parameter ratio = 13.5.

In the title compound, $C_{12}H_{16}N_4O_2$, the dihedral angle between the two pyrazole rings is 78.17 (7)°. Intermolecular O—H···N hydrogen bonds link the molecules into one-dimensional chains along the c axis.

Related literature

For the synthesis of bis(3,5-dimethylpyrazol-1-yl)acetic acid, see: Otero *et al.* (2004).



Experimental

Crystal data

$C_{12}H_{16}N_4O_2$
M_r = 248.29
Monoclinic, $P2_1/c$
a = 8.4317 (8) Å
b = 18.8569 (16) Å
c = 8.6083 (7) Å
β = 114.576 (7)°
V = 1244.69 (19) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 (2) K
0.50 × 0.40 × 0.35 mm

Data collection

Enraf–Nonius CAD-4 four-circle diffractometer
Absorption correction: none
2526 measured reflections
2305 independent reflections
1924 reflections with $I > 2\sigma(I)$
R_{int} = 0.007
3 standard reflections
frequency: 60 min
intensity decay: 0.2%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.120
S = 1.08
2305 reflections
171 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.23 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
O2—H2···N4^{i}	0.97 (3)	1.71 (3)	2.676 (2)	172 (3)

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD* (McArdle, 1999); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KJ2077).

References

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Otero, A., Fernández-Baeza, J., Antiñolo, A., Tejeda, J., Lara-Sánchez, A., Sánchez-Barba, L. & Rodríguez, A. M. (2004). *Eur. J. Inorg. Chem.* pp. 260–266.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

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supporting information

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Bis(3,5-dimethylpyrazol-1-yl)acetic acid

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S1. Comment

The title compound, $C_{12}H_{16}N_4O_2$, was synthesized through the protonation of bis(3,5-dimethylpyrazol-1-yl)acetate. This compound was used to prepare 2,2-bis(3,5-dimethylpyrazol-1-yl)ethanol, which was the reagent for the NNO monoanionic heteroscorpionate ligand (Otero *et al.*, 2004). Intermolecular O—H···N hydrogen bonds link the molecules into one-dimensional chains along the *c* axis (Fig. 2 and Table 1). The dihedral angle between the two pyrazol rings is 78.17 (7)°.

S2. Experimental

The title compound was synthesized according to the literature procedure (Otero *et al.*, 2004). Single crystals of the compound suitable for X-ray analysis were obtained by diffusion of ether into a THF solution.

S3. Refinement

All H-atoms except the H atom of the acid group, which was refined isotropically, were positioned geometrically and included in the refinement using a riding model with C—H = 0.98 Å, U_{iso}(H) = 1.2U_{eq}(C) for C(sp^3)—H, C—H = 0.96 Å, U_{iso}(H) = 1.5U_{eq}(C) for CH_3, and C—H = 0.93 Å, U_{iso}(H) = 1.2U_{eq}(C) for C(sp^2)—H.

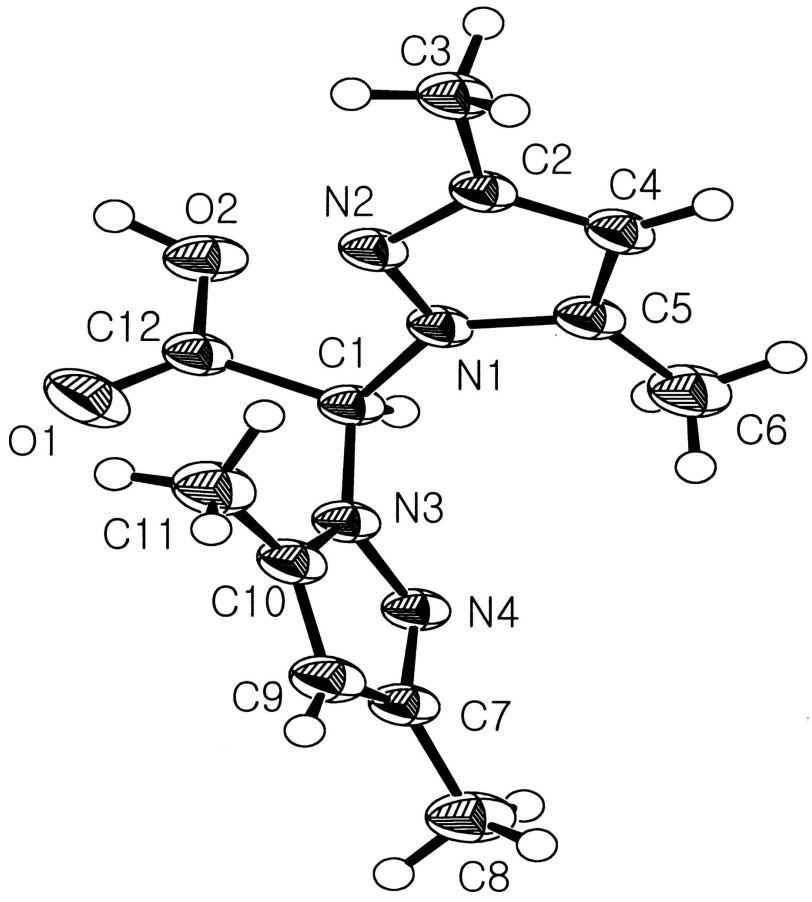

Figure 1

A view of the title compound. Displacement ellipsoids are drawn at the 40% probability level.

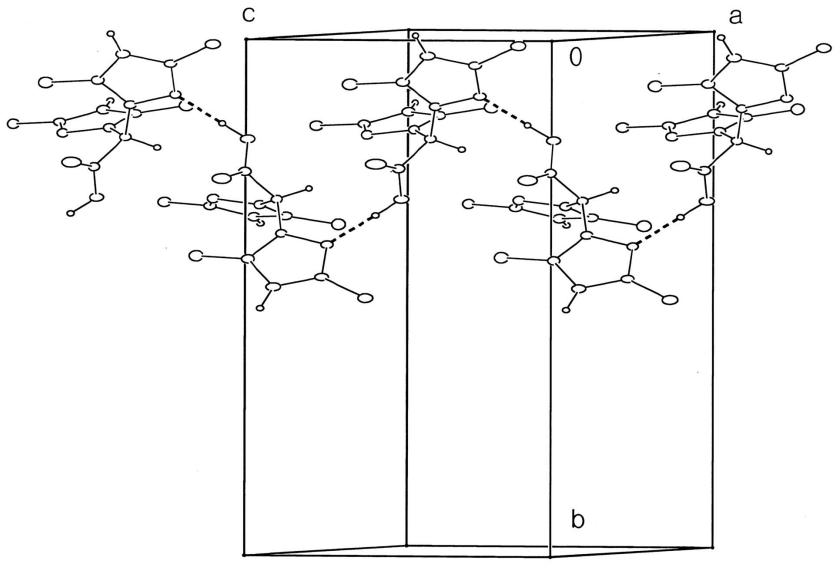

Figure 2

A packing diagram of the title compound viewed approximately perpendicular to the *bc* plane. Hydrogen bonds are indicated by dashed lines. H atoms of the methyl groups were omitted for clarity.

Bis(3,5-dimethylpyrazol-1-yl)acetic acid

Crystal data

$C_{12}H_{16}N_4O_2$
$M_r = 248.29$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 8.4317\ (8)$ Å
$b = 18.8569\ (16)$ Å
$c = 8.6083\ (7)$ Å
$\beta = 114.576\ (7)°$
$V = 1244.69\ (19)$ Å^3
$Z = 4$
$F(000) = 528$
$D_x = 1.325$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9.3–11.6°$
$\mu = 0.09$ mm^{-1}
$T = 293$ K
Block, colourless
0.50 × 0.40 × 0.35 mm

Data collection

Enraf–Nonius CAD-4 four-circle diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
2526 measured reflections
2305 independent reflections
1924 reflections with $I > 2\sigma(I)$
$R_{int} = 0.007$
$\theta_{max} = 25.5°$, $\theta_{min} = 2.8°$
$h = 0\rightarrow10$
$k = 0\rightarrow22$
$l = -10\rightarrow9$
3 standard reflections every 60 min
intensity decay: 0.2%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.120$
$S = 1.08$
2305 reflections
171 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0687P)^2 + 0.2835P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.01420 (18)	0.19070 (8)	0.40486 (18)	0.0328 (3)
H1A	0.0399	0.2115	0.3135	0.039*
N1	0.17999 (15)	0.17643 (7)	0.54704 (15)	0.0337 (3)
N2	0.19887 (16)	0.18728 (7)	0.71087 (15)	0.0363 (3)
C2	0.36285 (19)	0.16958 (8)	0.80788 (19)	0.0342 (3)
C3	0.4348 (2)	0.17514 (10)	0.9979 (2)	0.0444 (4)
H3A	0.3425	0.1868	1.0313	0.067*
H3B	0.5223	0.2115	1.0366	0.067*
H3C	0.4858	0.1306	1.0481	0.067*
C4	0.4481 (2)	0.14729 (9)	0.7074 (2)	0.0389 (4)
H4A	0.5633	0.1323	0.7462	0.047*
C5	0.3290 (2)	0.15185 (8)	0.5408 (2)	0.0359 (4)
C6	0.3447 (2)	0.13593 (11)	0.3783 (2)	0.0521 (5)
H6A	0.2877	0.0918	0.3328	0.078*
H6B	0.4657	0.1326	0.4000	0.078*
H6C	0.2908	0.1732	0.2975	0.078*
N3	−0.08641 (16)	0.12648 (7)	0.33639 (15)	0.0332 (3)
N4	−0.10877 (16)	0.10578 (7)	0.17637 (15)	0.0341 (3)
C7	−0.1927 (2)	0.04440 (8)	0.1485 (2)	0.0380 (4)
C8	−0.2378 (3)	0.00510 (10)	−0.0153 (2)	0.0554 (5)
H8A	−0.1900	0.0296	−0.0838	0.083*
H8B	−0.3623	0.0023	−0.0763	0.083*
H8C	−0.1900	−0.0419	0.0089	0.083*
C9	−0.2243 (2)	0.02556 (9)	0.2895 (2)	0.0435 (4)
H9A	−0.2817	−0.0150	0.3004	0.052*
C10	−0.15508 (19)	0.07794 (8)	0.4088 (2)	0.0379 (4)
C11	−0.1488 (3)	0.08317 (11)	0.5835 (2)	0.0524 (5)
H11A	−0.0345	0.0981	0.6619	0.079*
H11B	−0.1743	0.0377	0.6178	0.079*
H11C	−0.2335	0.1171	0.5838	0.079*
C12	−0.0880 (2)	0.24644 (8)	0.45592 (19)	0.0379 (4)
O1	−0.22573 (17)	0.23504 (8)	0.4587 (2)	0.0652 (4)
O2	−0.00521 (16)	0.30749 (6)	0.49036 (15)	0.0442 (3)
H2	−0.052 (3)	0.3382 (14)	0.551 (3)	0.081 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0339 (8)	0.0349 (8)	0.0287 (7)	−0.0003 (6)	0.0123 (6)	0.0000 (6)
N1	0.0312 (6)	0.0405 (7)	0.0293 (6)	0.0031 (5)	0.0126 (5)	−0.0017 (5)
N2	0.0350 (7)	0.0436 (7)	0.0308 (6)	0.0018 (5)	0.0141 (5)	−0.0015 (5)
C2	0.0332 (8)	0.0334 (7)	0.0345 (8)	−0.0007 (6)	0.0126 (6)	0.0020 (6)
C3	0.0430 (9)	0.0520 (10)	0.0347 (8)	0.0046 (7)	0.0127 (7)	0.0033 (7)
C4	0.0326 (8)	0.0428 (8)	0.0412 (8)	0.0068 (6)	0.0154 (6)	0.0044 (7)
C5	0.0360 (8)	0.0351 (8)	0.0395 (8)	0.0031 (6)	0.0187 (7)	0.0009 (6)
C6	0.0519 (10)	0.0673 (12)	0.0419 (9)	0.0139 (9)	0.0242 (8)	−0.0018 (8)
N3	0.0350 (7)	0.0345 (7)	0.0312 (6)	−0.0013 (5)	0.0149 (5)	0.0007 (5)
N4	0.0360 (7)	0.0339 (7)	0.0306 (6)	−0.0017 (5)	0.0121 (5)	−0.0009 (5)
C7	0.0373 (8)	0.0312 (7)	0.0394 (8)	−0.0002 (6)	0.0100 (6)	0.0009 (6)
C8	0.0637 (12)	0.0465 (10)	0.0476 (10)	−0.0099 (9)	0.0147 (9)	−0.0102 (8)
C9	0.0408 (9)	0.0371 (8)	0.0502 (10)	−0.0053 (7)	0.0165 (7)	0.0064 (7)
C10	0.0333 (8)	0.0412 (9)	0.0399 (8)	0.0027 (6)	0.0160 (6)	0.0077 (6)
C11	0.0513 (10)	0.0659 (12)	0.0472 (10)	−0.0005 (9)	0.0277 (8)	0.0084 (8)
C12	0.0355 (8)	0.0407 (8)	0.0332 (8)	0.0044 (6)	0.0101 (6)	−0.0002 (6)
O1	0.0395 (7)	0.0564 (8)	0.1023 (12)	0.0023 (6)	0.0320 (7)	−0.0173 (8)
O2	0.0562 (7)	0.0363 (6)	0.0463 (6)	0.0003 (5)	0.0276 (6)	−0.0049 (5)

Geometric parameters (Å, º)

C1—N1	1.4496 (18)	N3—C10	1.3644 (19)
C1—N3	1.4551 (19)	N3—N4	1.3673 (17)
C1—C12	1.535 (2)	N4—C7	1.325 (2)
C1—H1A	0.9800	C7—C9	1.392 (2)
N1—C5	1.3608 (19)	C7—C8	1.496 (2)
N1—N2	1.3673 (17)	C8—H8A	0.9600
N2—C2	1.327 (2)	C8—H8B	0.9600
C2—C4	1.400 (2)	C8—H8C	0.9600
C2—C3	1.493 (2)	C9—C10	1.369 (2)
C3—H3A	0.9600	C9—H9A	0.9300
C3—H3B	0.9600	C10—C11	1.486 (2)
C3—H3C	0.9600	C11—H11A	0.9600
C4—C5	1.369 (2)	C11—H11B	0.9600
C4—H4A	0.9300	C11—H11C	0.9600
C5—C6	1.489 (2)	C12—O1	1.1910 (19)
C6—H6A	0.9600	C12—O2	1.315 (2)
C6—H6B	0.9600	O2—H2	0.97 (3)
C6—H6C	0.9600		
N1—C1—N3	112.50 (12)	H6B—C6—H6C	109.5
N1—C1—C12	110.15 (11)	C10—N3—N4	111.21 (12)
N3—C1—C12	112.56 (12)	C10—N3—C1	131.29 (12)
N1—C1—H1A	107.1	N4—N3—C1	117.30 (11)
N3—C1—H1A	107.1	C7—N4—N3	105.74 (12)

C12—C1—H1A	107.1	N4—C7—C9	110.26 (14)
C5—N1—N2	112.22 (12)	N4—C7—C8	120.71 (15)
C5—N1—C1	127.72 (12)	C9—C7—C8	129.02 (15)
N2—N1—C1	120.06 (11)	C7—C8—H8A	109.5
C2—N2—N1	104.80 (12)	C7—C8—H8B	109.5
N2—C2—C4	110.80 (13)	H8A—C8—H8B	109.5
N2—C2—C3	120.94 (14)	C7—C8—H8C	109.5
C4—C2—C3	128.26 (14)	H8A—C8—H8C	109.5
C2—C3—H3A	109.5	H8B—C8—H8C	109.5
C2—C3—H3B	109.5	C10—C9—C7	107.06 (14)
H3A—C3—H3B	109.5	C10—C9—H9A	126.5
C2—C3—H3C	109.5	C7—C9—H9A	126.5
H3A—C3—H3C	109.5	N3—C10—C9	105.72 (14)
H3B—C3—H3C	109.5	N3—C10—C11	125.07 (15)
C5—C4—C2	106.67 (14)	C9—C10—C11	129.20 (15)
C5—C4—H4A	126.7	C10—C11—H11A	109.5
C2—C4—H4A	126.7	C10—C11—H11B	109.5
N1—C5—C4	105.51 (13)	H11A—C11—H11B	109.5
N1—C5—C6	123.35 (14)	C10—C11—H11C	109.5
C4—C5—C6	131.13 (15)	H11A—C11—H11C	109.5
C5—C6—H6A	109.5	H11B—C11—H11C	109.5
C5—C6—H6B	109.5	O1—C12—O2	125.75 (15)
H6A—C6—H6B	109.5	O1—C12—C1	123.40 (15)
C5—C6—H6C	109.5	O2—C12—C1	110.83 (13)
H6A—C6—H6C	109.5	C12—O2—H2	110.3 (15)

N3—C1—N1—C5	78.86 (18)	N1—C1—N3—N4	−111.63 (13)
C12—C1—N1—C5	−154.68 (15)	C12—C1—N3—N4	123.21 (13)
N3—C1—N1—N2	−100.73 (15)	C10—N3—N4—C7	0.43 (16)
C12—C1—N1—N2	25.73 (18)	C1—N3—N4—C7	175.80 (12)
C5—N1—N2—C2	0.43 (17)	N3—N4—C7—C9	−0.02 (16)
C1—N1—N2—C2	−179.92 (13)	N3—N4—C7—C8	−179.15 (14)
N1—N2—C2—C4	−0.27 (17)	N4—C7—C9—C10	−0.39 (18)
N1—N2—C2—C3	179.31 (14)	C8—C7—C9—C10	178.65 (16)
N2—C2—C4—C5	0.02 (19)	N4—N3—C10—C9	−0.66 (17)
C3—C2—C4—C5	−179.52 (15)	C1—N3—C10—C9	−175.19 (14)
N2—N1—C5—C4	−0.42 (17)	N4—N3—C10—C11	178.27 (14)
C1—N1—C5—C4	179.97 (14)	C1—N3—C10—C11	3.7 (3)
N2—N1—C5—C6	−179.67 (15)	C7—C9—C10—N3	0.62 (18)
C1—N1—C5—C6	0.7 (2)	C7—C9—C10—C11	−178.25 (16)
C2—C4—C5—N1	0.24 (17)	N1—C1—C12—O1	−117.82 (17)
C2—C4—C5—C6	179.40 (17)	N3—C1—C12—O1	8.6 (2)
N1—C1—N3—C10	62.62 (19)	N1—C1—C12—O2	63.93 (16)
C12—C1—N3—C10	−62.5 (2)	N3—C1—C12—O2	−169.64 (12)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···N4^{i}	0.97 (3)	1.71 (3)	2.676 (2)	172 (3)

Symmetry code: (i) x, $-y+1/2$, $z+1/2$.