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Crystal structure of ethyl 4-[(1*H*-pyrazol-1-yl)methyl]benzoate

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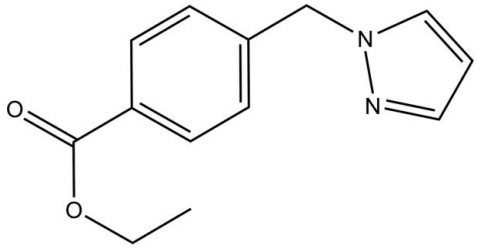
In the title molecule, $C_{13}H_{14}N_2O_2$, the dihedral angle between the pyrazole and benzene ring mean planes is 76.06 (11)°, and the conformation of the ethyl side chain is *anti* [C—O—C—C = −175.4 (3)°]. In the crystal, the only directional interactions are very weak C—H $\cdots\pi$ interactions involving both the pyrazole and benzene rings, leading to the formation of a three-dimensional network.

Keywords: crystal structure; ester; pyrazole derivative.

CCDC reference: 1034364

1. Related literature

For a related structure, see: Dong *et al.* (2011). For background to the properties of pyrazole derivatives, see: Adnan & Tarek (2004); Ashraf *et al.* (2003).



2. Experimental

2.1. Crystal data

$C_{13}H_{14}N_2O_2$
M_r = 230.26
Triclinic, $P\overline{1}$
a = 8.1338 (12) Å
b = 8.1961 (9) Å
c = 10.7933 (11) Å
α = 74.013 (9)°
β = 83.308 (10)°
γ = 64.734 (13)°
V = 625.54 (13) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 293 K
0.22 × 0.20 × 0.18 mm

2.2. Data collection

Agilent SuperNova (Single source at offset, Eos) diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Agilent, 2012)
T_{min} = 0.982, T_{max} = 0.985
4295 measured reflections
2197 independent reflections
1639 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.201
S = 1.15
2197 reflections
155 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

*Cg*1 and *Cg*2 are the centroids of the N1/N2/C1–C3 and C5–C10 rings, respectively.

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C2—H2$\cdots Cg2^{i}$	0.93	2.94	3.670 (4)	137
C4—H4$A\cdots Cg1^{ii}$	0.97	3.00	3.600 (3)	122
C12—H12$A\cdots Cg2^{iii}$	0.97	2.82	3.689 (4)	150

Symmetry codes: (i) $x-1, y, z$; (ii) $-x, -y, -z$; (iii) $-x, -y+1, -z+1$.

Data collection: *FRAMBO* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7319).

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supporting information

Acta Cryst. (2014). E**70**, o1287 [doi:10.1107/S1600536814025100]

Crystal structure of ethyl 4-[(1*H*-pyrazol-1-yl)methyl]benzoate

Ju-Xian Wang and Chao Feng

S1. Comment

Pyrazole and its derivatives are an important class of *N*-heterocyclic compounds because they exhibit a broad spectrum of pharmacological activities such as antifungal (Adnan & Tarek, 2004), antitumor and antiangiogenic activities (Ashraf *et al.*, 2003). As part of pyrazole derivatives, the crystal structure of 1, 4-bis[(1*H*-pyrazol-1-yl)methyl]benzene has been determined (Dong *et al.*, 2011). As part of this ongoing search for new pyrazole compounds, the title compound was synthesized and its crystal structure is reported herein. In the title compound (Fig. 1), bond lengths and angles fall in normal ranges. The dihedral angle between the pyrazole ring (N1/N2/C1—C3) and the phenyl ring (C5—C10) is 76.06 (11) °. In the crystal, there exists weak C–H···π contacts.

S2. Experimental

In a 250 ml four-necked round-bottom flask equipped with a mechanical stirrer, pyrazole (0.680 g 10 mmol), potassium carbonate (2.073 g 15 mmol) and 1-(4-(bromomethyl)phenyl)-1-hydroxypentan-2-one (2.712 g, 10 mmol) were cautiously dissolved in acetone (100 ml). The solution was heated at 65 °C for 6 h, then the mixture was filtered off was removed by rotatory evaporator at 35 °C and the crude product was obtained 1.815 g (66.1%). Colourless blocks of the title compound were obtained from ethanol by slow evaporation.

S3. Refinement

H-atoms were placed in calculated positions and refined constrained to ride on their parent atoms, with C—H = 0.93–0.97 Å, U_{iso}(H) = 1.5U_{eq}(C) for methyl and 1.2U_{eq}(C) for the methylene.

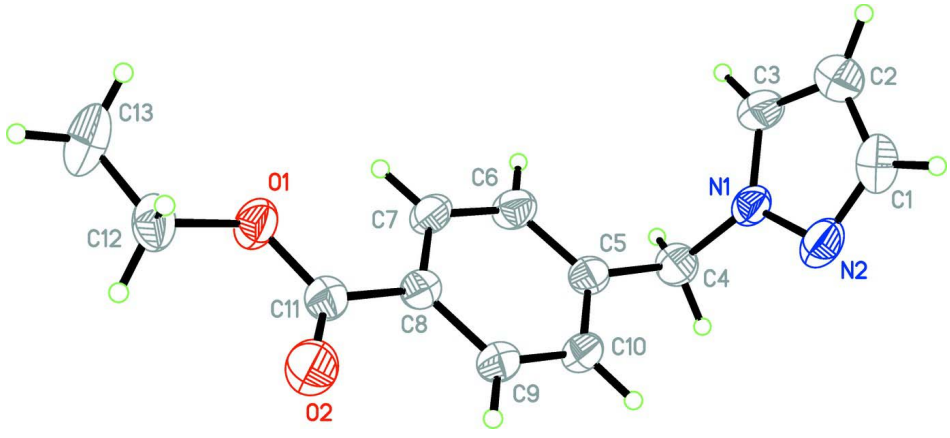

Figure 1

The molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level.

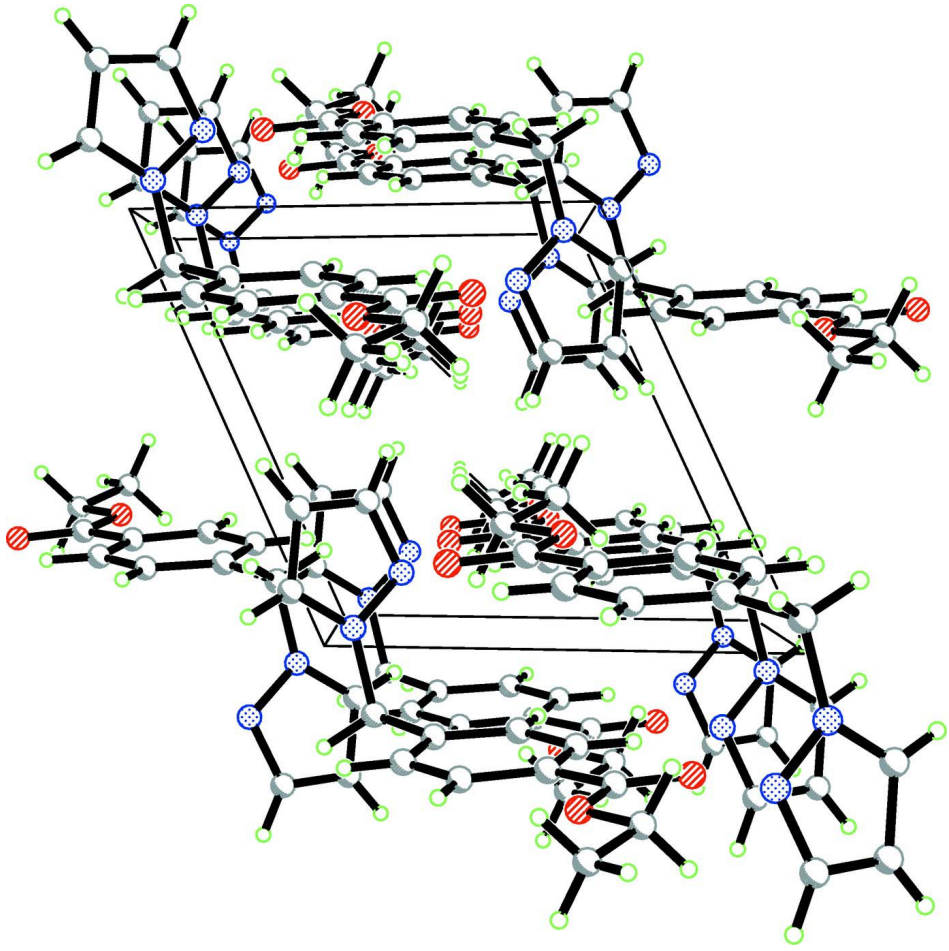

Figure 2

View of the packing diagram of the title compound along the *b* axis.

Ethyl 4-[(1*H*-pyrazol-1-yl)methyl]benzoate

Crystal data

$C_{13}H_{14}N_2O_2$
$M_r = 230.26$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 8.1338$ (12) Å
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$c = 10.7933$ (11) Å
$\alpha = 74.013$ (9)°
$\beta = 83.308$ (10)°
$\gamma = 64.734$ (13)°
$V = 625.54$ (13) Å^3
$Z = 2$
$F(000) = 244$
$D_x = 1.222$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1485 reflections
$\theta = 4.3$–28.2°
$\mu = 0.08$ mm^{-1}
$T = 293$ K
Block, colourless
0.22 × 0.20 × 0.18 mm

Data collection

Agilent SuperNova (Single source at offset, Eos) diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*CrysAlis RED*; Agilent, 2012)

T_{min} = 0.982, T_{max} = 0.985
4295 measured reflections
2197 independent reflections
1639 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

θ_{max} = 25.0°, θ_{min} = 3.0°
h = −9→9
k = −9→9
l = −12→8

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.201
S = 1.15
2197 reflections
155 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
$w = 1/[\sigma^2(F_o^2) + (0.079P)^2 + 0.2197P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
C1	−0.3321 (5)	0.2354 (4)	0.0922 (3)	0.0772 (9)
H1	−0.4412	0.3219	0.0516	0.093*
C2	−0.3185 (4)	0.0903 (5)	0.1964 (3)	0.0787 (9)
H2	−0.4119	0.0606	0.2394	0.094*
C3	−0.1386 (4)	−0.0001 (4)	0.2228 (3)	0.0718 (9)
H3	−0.0841	−0.1068	0.2885	0.086*
C4	0.1384 (4)	0.0519 (4)	0.1369 (3)	0.0653 (8)
H4A	0.1766	0.0861	0.0486	0.078*
H4B	0.2091	−0.0805	0.1712	0.078*
C5	0.1753 (3)	0.1603 (4)	0.2163 (2)	0.0543 (7)
C6	0.2157 (4)	0.0834 (4)	0.3464 (3)	0.0610 (7)
H6	0.2269	−0.0374	0.3842	0.073*
C7	0.2396 (4)	0.1856 (4)	0.4205 (2)	0.0584 (7)
H7	0.2671	0.1326	0.5077	0.070*
C8	0.2227 (3)	0.3657 (3)	0.3656 (2)	0.0505 (6)
C9	0.1870 (4)	0.4413 (4)	0.2343 (3)	0.0573 (7)
H9	0.1784	0.5611	0.1959	0.069*
C10	0.1643 (4)	0.3379 (4)	0.1609 (3)	0.0588 (7)
H10	0.1413	0.3888	0.0731	0.071*
C11	0.2393 (4)	0.4838 (4)	0.4420 (3)	0.0561 (7)

C12	0.2906 (4)	0.5017 (5)	0.6500 (3)	0.0726 (9)
H12A	0.1747	0.6050	0.6552	0.087*
H12B	0.3805	0.5512	0.6164	0.087*
C13	0.3460 (6)	0.3720 (6)	0.7790 (4)	0.1028 (13)
H13A	0.2574	0.3219	0.8103	0.154*
H13B	0.3544	0.4378	0.8374	0.154*
H13C	0.4622	0.2722	0.7729	0.154*
N1	−0.0526 (3)	0.0895 (3)	0.13866 (19)	0.0554 (6)
N2	−0.1716 (4)	0.2383 (3)	0.0558 (2)	0.0741 (8)
O1	0.2758 (3)	0.3958 (3)	0.56635 (18)	0.0672 (6)
O2	0.2213 (3)	0.6418 (3)	0.3986 (2)	0.0823 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.075 (2)	0.072 (2)	0.083 (2)	−0.0205 (17)	−0.0233 (17)	−0.0225 (17)
C2	0.070 (2)	0.088 (2)	0.079 (2)	−0.0365 (18)	0.0043 (16)	−0.0178 (18)
C3	0.078 (2)	0.075 (2)	0.0547 (17)	−0.0352 (17)	−0.0020 (14)	0.0029 (15)
C4	0.0684 (18)	0.083 (2)	0.0557 (17)	−0.0358 (16)	0.0119 (13)	−0.0318 (15)
C5	0.0541 (15)	0.0664 (17)	0.0469 (15)	−0.0276 (13)	0.0101 (11)	−0.0209 (12)
C6	0.0804 (19)	0.0549 (16)	0.0506 (16)	−0.0330 (14)	0.0032 (13)	−0.0109 (12)
C7	0.0788 (19)	0.0601 (16)	0.0391 (14)	−0.0336 (14)	0.0003 (12)	−0.0086 (12)
C8	0.0534 (14)	0.0530 (14)	0.0456 (14)	−0.0235 (12)	0.0016 (11)	−0.0114 (11)
C9	0.0660 (17)	0.0557 (15)	0.0489 (15)	−0.0289 (13)	−0.0030 (12)	−0.0035 (12)
C10	0.0630 (17)	0.0721 (18)	0.0404 (14)	−0.0305 (14)	0.0011 (11)	−0.0090 (13)
C11	0.0593 (16)	0.0558 (16)	0.0552 (16)	−0.0262 (13)	−0.0016 (12)	−0.0124 (13)
C12	0.079 (2)	0.082 (2)	0.072 (2)	−0.0358 (17)	−0.0023 (15)	−0.0388 (17)
C13	0.131 (3)	0.105 (3)	0.070 (2)	−0.031 (2)	−0.027 (2)	−0.039 (2)
N1	0.0692 (14)	0.0607 (13)	0.0401 (12)	−0.0296 (11)	−0.0015 (10)	−0.0136 (10)
N2	0.100 (2)	0.0673 (16)	0.0533 (14)	−0.0374 (14)	−0.0200 (13)	−0.0007 (12)
O1	0.0917 (15)	0.0665 (12)	0.0502 (12)	−0.0353 (11)	−0.0060 (10)	−0.0181 (9)
O2	0.1173 (19)	0.0621 (13)	0.0756 (15)	−0.0452 (13)	−0.0147 (13)	−0.0102 (11)

Geometric parameters (Å, º)

C1—N2	1.327 (4)	C7—H7	0.9300
C1—C2	1.367 (5)	C8—C9	1.391 (3)
C1—H1	0.9300	C8—C11	1.487 (4)
C2—C3	1.351 (4)	C9—C10	1.385 (4)
C2—H2	0.9300	C9—H9	0.9300
C3—N1	1.333 (4)	C10—H10	0.9300
C3—H3	0.9300	C11—O2	1.200 (3)
C4—N1	1.448 (3)	C11—O1	1.336 (3)
C4—C5	1.524 (4)	C12—O1	1.460 (3)
C4—H4A	0.9700	C12—C13	1.481 (5)
C4—H4B	0.9700	C12—H12A	0.9700
C5—C10	1.381 (4)	C12—H12B	0.9700
C5—C6	1.385 (4)	C13—H13A	0.9600

C6—C7	1.386 (4)	C13—H13B	0.9600
C6—H6	0.9300	C13—H13C	0.9600
C7—C8	1.384 (4)	N1—N2	1.348 (3)
N2—C1—C2	112.2 (3)	C10—C9—C8	119.9 (2)
N2—C1—H1	123.9	C10—C9—H9	120.1
C2—C1—H1	123.9	C8—C9—H9	120.1
C3—C2—C1	104.4 (3)	C5—C10—C9	120.9 (2)
C3—C2—H2	127.8	C5—C10—H10	119.5
C1—C2—H2	127.8	C9—C10—H10	119.5
N1—C3—C2	108.4 (3)	O2—C11—O1	122.7 (3)
N1—C3—H3	125.8	O2—C11—C8	124.4 (3)
C2—C3—H3	125.8	O1—C11—C8	112.9 (2)
N1—C4—C5	111.3 (2)	O1—C12—C13	107.1 (3)
N1—C4—H4A	109.4	O1—C12—H12A	110.3
C5—C4—H4A	109.4	C13—C12—H12A	110.3
N1—C4—H4B	109.4	O1—C12—H12B	110.3
C5—C4—H4B	109.4	C13—C12—H12B	110.3
H4A—C4—H4B	108.0	H12A—C12—H12B	108.5
C10—C5—C6	119.0 (2)	C12—C13—H13A	109.5
C10—C5—C4	120.7 (2)	C12—C13—H13B	109.5
C6—C5—C4	120.3 (2)	H13A—C13—H13B	109.5
C5—C6—C7	120.4 (2)	C12—C13—H13C	109.5
C5—C6—H6	119.8	H13A—C13—H13C	109.5
C7—C6—H6	119.8	H13B—C13—H13C	109.5
C8—C7—C6	120.4 (2)	C3—N1—N2	110.7 (3)
C8—C7—H7	119.8	C3—N1—C4	127.7 (2)
C6—C7—H7	119.8	N2—N1—C4	121.4 (2)
C7—C8—C9	119.2 (2)	C1—N2—N1	104.3 (2)
C7—C8—C11	122.5 (2)	C11—O1—C12	117.0 (2)
C9—C8—C11	118.3 (2)		
N2—C1—C2—C3	−0.8 (4)	C7—C8—C11—O2	−178.2 (3)
C1—C2—C3—N1	0.6 (4)	C9—C8—C11—O2	1.1 (4)
N1—C4—C5—C10	−88.7 (3)	C7—C8—C11—O1	1.5 (4)
N1—C4—C5—C6	89.5 (3)	C9—C8—C11—O1	−179.2 (2)
C10—C5—C6—C7	1.8 (4)	C2—C3—N1—N2	−0.3 (4)
C4—C5—C6—C7	−176.5 (2)	C2—C3—N1—C4	174.4 (3)
C5—C6—C7—C8	0.2 (4)	C5—C4—N1—C3	−89.3 (4)
C6—C7—C8—C9	−2.0 (4)	C5—C4—N1—N2	84.9 (3)
C6—C7—C8—C11	177.3 (2)	C2—C1—N2—N1	0.6 (4)
C7—C8—C9—C10	1.6 (4)	C3—N1—N2—C1	−0.2 (3)
C11—C8—C9—C10	−177.7 (2)	C4—N1—N2—C1	−175.3 (2)
C6—C5—C10—C9	−2.1 (4)	O2—C11—O1—C12	1.0 (4)
C4—C5—C10—C9	176.1 (2)	C8—C11—O1—C12	−178.7 (2)
C8—C9—C10—C5	0.4 (4)	C13—C12—O1—C11	−175.4 (3)

Hydrogen-bond geometry (Å, º)

Cg1 and Cg2 are the centroids of the N1/N2/C1–C3 and C5–C10 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···*Cg*2^{i}	0.93	2.94	3.670 (4)	137
C4—H4*A*···*Cg*1^{ii}	0.97	3.00	3.600 (3)	122
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