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Ethyl 3-[(1,5-dimethylpyrazol-4-yl)carbonylhydrazino]butyrate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.051
wR factor = 0.156
Data-to-parameter ratio = 14.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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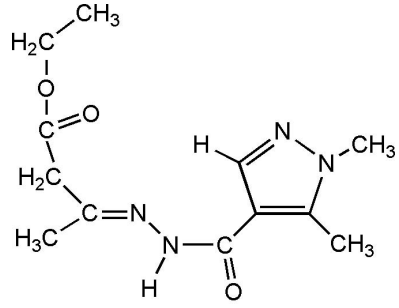
In the crystal structure of the title compound, $C_{12}H_{18}N_4O_3$, the molecules interact through intermolecular N—H···O and C—H···O hydrogen bonds to form dimers, providing stabilization.

Comment

Pyrazole and its derivatives represent one of the most active classes of compounds, possessing a wide spectrum of biological activities (Makino, *et al.*,1999), such as antibacterial, fungicidal (Chen *et al.*, 2000), herbicidal (Krishnaiah & Narsaiah, 2002) and insecticidal (Huang, *et al.*, 1996). In the course of our systematic studies aimed at the synthesis of new bioactive compounds, the title compound, (I), was obtained accidentally; its structure is reported here.



(I)

The bond distances and angles (Table 1) are as expected for this type of compound. In the crystal structure, centro-

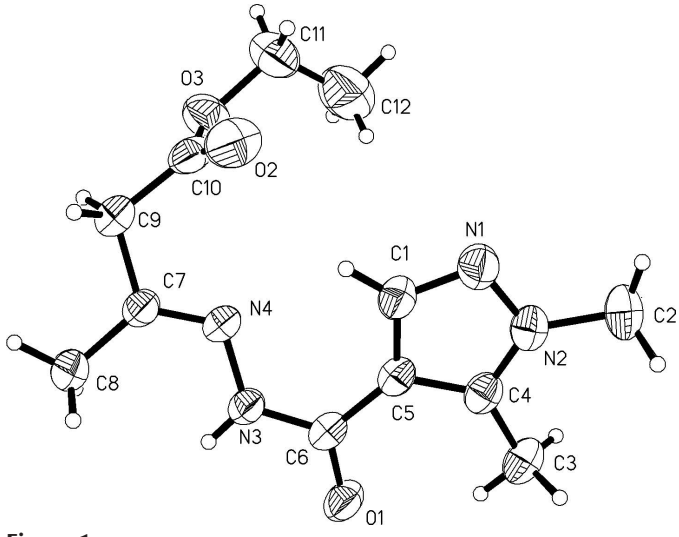


Figure 1
View of the title compound, with 35% probability displacement ellipsoids.

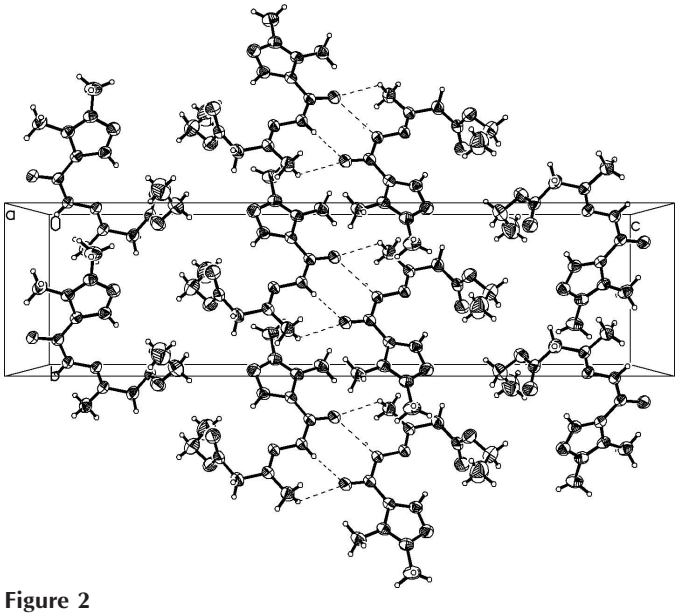


Figure 2
The molecular packing of the title compound viewed along the *a* axis. Hydrogen bonds are shown as dashed lines.

symmetrically related molecules are linked into dimers by intermolecular N—H···O and C—H···O hydrogen interactions (Table 2 and Fig. 2), thus generating rings of graph-set motifs $R_2^1(7)$ and $R_2^2(8)$ (Bernstein *et al.*, 1995).

Experimental

Ethyl 1,5-dimethyl-1*H*-pyrazole-4-carboxylate, (1), was synthesized according to the procedure published by Beck & Lynch (1987). 1,5-Dimethyl-1*H*-pyrazole-4-carbohydrazide, (2), was prepared by the reaction of hydrazine hydrate with (1). A mixture of (2) (2 mmol, 0.308 g) and ethyl acetoacetate (2 mmol, 0.260 g) in ethanol (20 ml) was refluxed for 4 h (monitored by thin-layer chromatography). The mixture was cooled and the title compound was obtained by filtration. Single crystals suitable for X-ray diffraction studies were isolated by recrystallization from ethanol.

Crystal data

$C_{12}H_{18}N_4O_3$
M_r = 266.30
Orthorhombic, *Pbca*
a = 11.5212 (18) Å
b = 7.9018 (12) Å
c = 30.551 (5) Å
V = 2781.3 (8) Å^3
Z = 8
D_x = 1.272 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2468 reflections
θ = 2.2–21.2°
μ = 0.09 mm^{-1}
T = 293 (2) K
Prism, colorless
0.48 × 0.42 × 0.18 mm

Data collection

Bruker APEX II CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.956, T_{max} = 0.983
18790 measured reflections
2452 independent reflections
1751 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 25.0°
$h = -10 \rightarrow 13$
$k = -9 \rightarrow 9$
$l = -36 \rightarrow 36$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.156
S = 0.99
2452 reflections
175 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0744P)^2 + 1.2421P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C1	1.311 (3)	N4—C7	1.274 (3)
N1—N2	1.359 (3)	C1—C5	1.403 (3)
N2—C4	1.336 (3)	C4—C5	1.396 (3)
N3—N4	1.388 (2)		
C1—N1—N2	104.3 (2)	N2—C4—C5	106.15 (19)
C4—N2—N1	113.01 (19)	C4—C5—C1	104.0 (2)
N1—C1—C5	112.5 (2)		

Table 2
Hydrogen-bond geometry (Å, °) .

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3D···O1^{i}	0.87 (2)	2.05 (2)	2.901 (3)	168 (2)
C8—H8A···O1^{i}	0.96	2.56	3.205 (3)	124

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

The H atom associated with N3 was located in a difference Fourier map and refined with the N—H distance restrained to 0.086 (2) Å and with U_{iso}(H) = 1.2U_{eq}(N). All other H atoms were placed in calculated positions, and included in the final cycles of refinement using a riding model (C—H = 0.93–0.97 Å), with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C_{methyl}).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

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