

Diethyl 1*H*-pyrazole-3,5-dicarboxylateCaixia Yin,^a Fangjun Huo,^b
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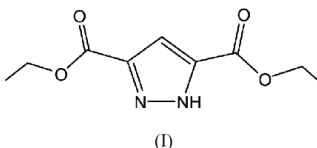
Key indicators

Single-crystal X-ray study
T = 298 K
Mean $\sigma(\text{C}-\text{C}) = 0.005 \text{ \AA}$
R factor = 0.047
wR factor = 0.113
Data-to-parameter ratio = 14.2For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The crystal structure of the title compound, $\text{C}_9\text{H}_{12}\text{N}_2\text{O}_4$, has been determined in the monoclinic space group $P2_1/c$. There are two molecules in the asymmetric unit and the two pyrazole rings are almost coplanar. The two molecules have different conformations and are connected into a dimer *via* intermolecular $\text{N}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds.

Comment

The title compound, diethyl 1*H*-pyrazole-3,5-dicarboxylate, (I), is very useful in organic synthesis. Such compounds are usually regarded as key synthons of oligomeric carboxamides, which are known to bind to DNA by lodging in the groove (Lee *et al.*, 1989; Chambers *et al.*, 1985). In addition, the title compound is also an intermediate for synthesizing polypodal ligands that are suited for the preparation of dimetallic complexes (Belle *et al.*, 1994).



Selected geometric parameters of (I) are listed in Table 1. Hydrogen bonding information is given in Table 2. The molecular conformation and crystal packing are illustrated in Figs. 1 and 2, respectively.

Experimental

The title compound was prepared according to the literature (Schenck *et al.*, 1985). An ethanol solution of pyrazole-3,5-dicarboxylic acid was treated with $\text{HCl}(\text{g})$ until the solution boiled. The solution was allowed to cool to room temperature and the HCl treatment repeated. After stirring overnight, the precipitate was filtered off and washed with ethanol. The combined filtrate and washings were evaporated under reduced pressure to yield a colorless oil (yield 78%). The oil was left at room temperature for several days and colorless crystals suitable for single-crystal X-ray diffraction study formed.

Crystal data

 $\text{C}_9\text{H}_{12}\text{N}_2\text{O}_4$
M_r = 212.21
Monoclinic, $P2_1/c$
a = 9.184 (2) \AA
b = 14.147 (3) \AA
c = 17.302 (4) \AA
 β = 100.074 (4)°
V = 2213.2 (9) \AA^3
Z = 8 $D_x = 1.274 \text{ Mg m}^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 10969 reflections
 $\theta = 2.3\text{--}18.9^\circ$
 $\mu = 0.10 \text{ mm}^{-1}$
T = 298 (2) K
Plate, colorless
0.30 × 0.30 × 0.10 mmReceived 6 January 2004
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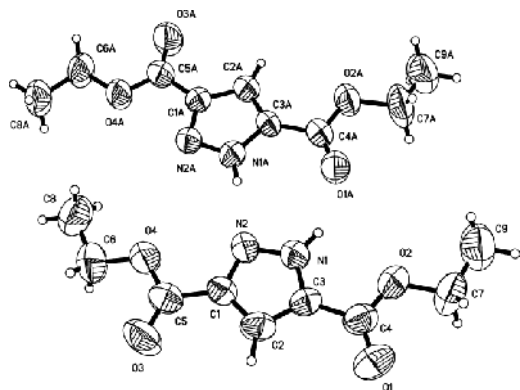


Figure 1
A view of the asymmetric unit of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

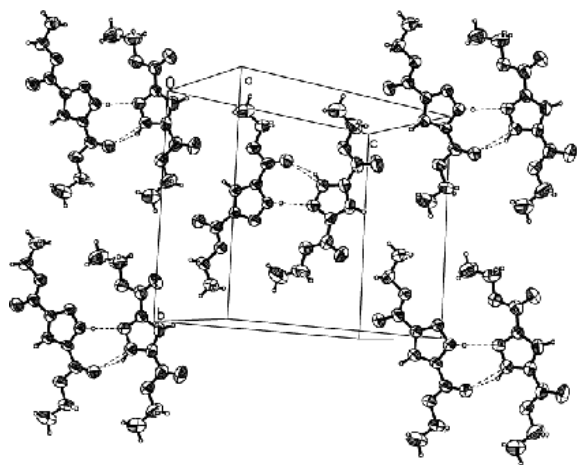


Figure 2
A packing diagram of the title compound.

Data collection

Bruker SMART CCD area-detector diffractometer	3892 independent reflections
ω scans	1424 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.061$
$T_{\text{min}} = 0.970$, $T_{\text{max}} = 0.990$	$\theta_{\text{max}} = 25.0^\circ$
8965 measured reflections	$h = -10 \rightarrow 10$
	$k = -16 \rightarrow 8$
	$l = -20 \rightarrow 20$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.047$	$w = 1/[\sigma^2(F_o^2) + (0.0355P)^2]$
$wR(F^2) = 0.113$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.81$	$(\Delta/\sigma)_{\text{max}} < 0.001$
3892 reflections	$\Delta\rho_{\text{max}} = 0.13 \text{ e } \text{\AA}^{-3}$
275 parameters	$\Delta\rho_{\text{min}} = -0.14 \text{ e } \text{\AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

N1—N2	1.335 (3)	N1A—N2A	1.332 (3)
C1—N2	1.333 (3)	C1A—N2A	1.340 (3)
C1—C2	1.393 (4)	C2A—C1A	1.394 (3)
C2—C3	1.361 (3)	C3A—C2A	1.359 (3)
C3—N1	1.349 (3)	C3A—N1A	1.359 (3)
C2—C3—C4	132.4 (3)	C2A—C3A—C4A	132.9 (3)
C2—C1—C5	128.1 (3)	C2A—C1A—C5A	127.4 (3)
O1—C4—C3	124.7 (4)	O1A—C4A—C3A	125.4 (3)
O2—C7—C9	108.7 (3)	O3A—C5A—C1A	123.0 (3)
O3—C5—C1	122.7 (4)	N1A—C3A—C4A	121.1 (3)
N1—C3—C4	122.0 (3)	C5A—O4A—C6A	116.5 (3)
N2—C1—C5	120.7 (3)	C4A—O2A—C7A	116.1 (3)
C4—O2—C7	117.9 (3)	N2A—C1A—C5A	121.3 (3)
C5—O4—C6	116.5 (3)	O4A—C6A—C8A	107.2 (3)

Table 2

Hydrogen-bonding geometry (\AA , $^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N1—H1 \cdots O1A	0.86	2.02	2.790 (3)	149
N1A—H1A \cdots N2	0.86	2.04	2.898 (3)	177

All H atoms were initially located in a difference Fourier map. All H atoms were treated as riding, with $Csp^3-H = 0.96-0.97 \text{ \AA}$, $Csp^2-H = 0.93 \text{ \AA}$ and $N-H = 0.86 \text{ \AA}$, and $U_{\text{iso}}(H) = 1.5U_{\text{eq}}(Csp^2, N)$ and $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(Csp^2, N)$.

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); 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, 2000); software used to prepare material for publication: SHELXTL.

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