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Diethyl pyridine-2,6-dicarboxylate

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.002 Å
R factor = 0.049
wR factor = 0.168
Data-to-parameter ratio = 15.5

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

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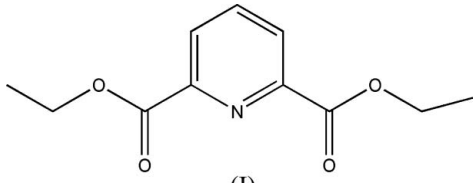
In the crystal structure of the title compound, $C_{11}H_{13}NO_4$, the asymmetric unit contains only one half-molecule; the molecule lies on a twofold rotation axis. The crystal structure is stabilized by intermolecular C—H···O and C—H···N hydrogen bonds, linking the molecules along the *b* axis.

Comment

The title compound is an effective material for preparing diacetylpyridine–hydrazine condensed macrocycle complexes (Yang *et al.*, 2002). We report here the crystal structure of the title compound, (I).



(I)

In the crystal structure of the title compound, (I) (Fig. 1), the asymmetric unit contains only one half-molecule. A twofold rotation axis passes through atoms N, C6 and H6*A*. The bond lengths and angles are within normal ranges (Allen *et al.*, 1987).

The central ring *A* (N/C4–C6/C4*A*/C5*A*) is, of course, planar and the O1/O2/C1–C4 group is nearly planar, with a puckering amplitude of Q_T = 0.0861 (3) Å (Cremer & Pople, 1975). The dihedral angle between these planes is 7.11 (3)°.

As can be seen from the packing diagram (Fig. 2), intermolecular C—H···O and C—H···N hydrogen bonds (Table 1) link the molecules along the *b* axis. Dipole–dipole and van der

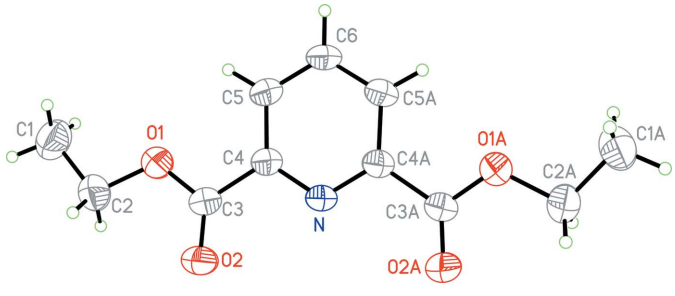


Figure 1
The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry code: (A) $-x, y, \frac{1}{2}-z$.]

Waals interactions are also effective in the molecular packing in the crystal structure.

Experimental

Pyridine-2,6-dicarbonylchloride (36.5 g) was placed in a three-necked flask fitted with a reflux condenser and absolute ethyl alcohol (300 cc) was added in small portions through the dropping funnel. The alcoholic solution was refluxed for 1 h and the excess alcohol was removed by distillation. The residue was cooled in an ice-bath, covered with a layer of diethyl ether, and a sufficient solution of sodium carbonate (20%) was added to make the aqueous layer alkaline. This layer was then separated and extracted twice with small portions of diethyl ether. The diethyl ether was removed from the combined extracts by distillation. Further distillation of the residue gave the title compound, which was crystallized from diethyl ether (yield 36.0 g, 90%; m.p. 315–316 K).

Crystal data

$C_{11}H_{13}NO_4$
M_r = 223.22
Monoclinic, $C2/c$
a = 15.222 (3) Å
b = 6.3480 (13) Å
c = 12.423 (3) Å
β = 96.45 (3)°
V = 1192.8 (5) Å^3
Z = 4
D_x = 1.243 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 296 (2) K
Block, colorless
0.40 × 0.30 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.964, T_{max} = 0.982
1226 measured reflections
1166 independent reflections
799 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 26.0°
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.168
S = 1.09
1166 reflections
75 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.019 (5)

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

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5A···O2^{ii}	0.93	2.55	3.212 (2)	128
C6—H6A···N^{ii}	0.93	2.62	3.552 (3)	180
C6—H6A···N^{iii}	0.93	2.62	3.552 (3)	180

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

H atoms were positioned geometrically, with C—H = 0.93, 0.97 and 0.96 Å for aromatic, methylene and methyl H atoms, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = xU_{eq}(C), where x = 1.5 for methyl H and x = 1.2 for other H.

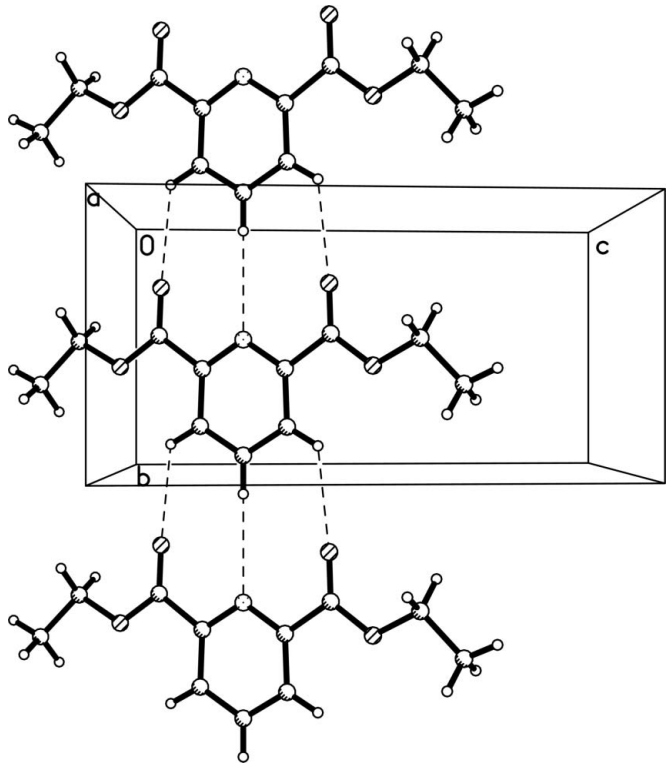


Figure 2
A packing diagram of (I). The intermolecular hydrogen bonds are shown as dashed lines.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1985); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); 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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