

Ping Du, Xiao-Ru Zhang,
Min-Hua Xu and
Shu-Sheng Zhang*College of Chemistry and Molecular
Engineering, Qingdao University of Science and
Technology, 266042 Qingdao, Shandong,
People's Republic of China

Correspondence e-mail: shushzhang@126.com

Key indicators

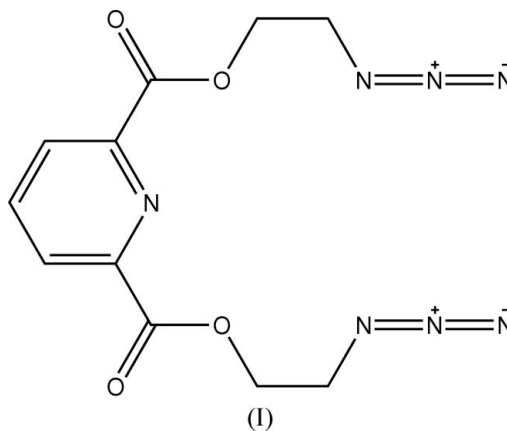
Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(C-C) = 0.002$ Å
 R factor = 0.048
 wR factor = 0.137
Data-to-parameter ratio = 14.0For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

Bis(2-azidoethyl) pyridine-2,6-dicarboxylate

The title compound, $C_{11}H_{11}N_7O_4$, possesses crystallographically imposed C_2 symmetry. The two side chains are twisted in opposite directions from the mean plane of the pyridine ring. $C-H \cdots O$ interactions link symmetry-related molecules into two-dimensional layers perpendicular to the a axis.

Comment

Azides are used to synthesize triazole or tetrazole derivatives, which frequently exhibit significant biological activity and, in consequence, have found a wide variety of applications as active heterocycles in pharmacy and agriculture (Ostrovskii & Koren, 2000; Nomiya *et al.*, 2000). In addition, photolysis of azides leads to the formation of azido radicals, which are employed industrially in the photoinitiation of radical polymerization (Endicott *et al.*, 1970). In an attempt to synthesize new triazoles or tetrazoles, the title compound, (I), was synthesized and its structure is reported here.



The asymmetric unit of (I) contains one half-molecule, the other half being related by a crystallographic twofold axis passing through the $C1 \cdots N4$ vector. The bond lengths and angles in (I) show normal values (Allen *et al.*, 1987). The molecule is not planar, the two side chains twisted in opposite directions from the mean plane of the pyridine ring. In the packing of the title compound, molecules are linked into two-dimensional layers perpendicular to the a axis (Fig. 2) by $C-H \cdots O$ intermolecular hydrogen bonds (Table 2). These interactions, together with van der Waals forces, stabilize the crystal structure.

Experimental

A mixture of pyridine-2,6-dicarboxylic acid (1.0 g, 6.0 mmol) and thionyl chloride (3 ml) was refluxed for 3 h. To this solution,

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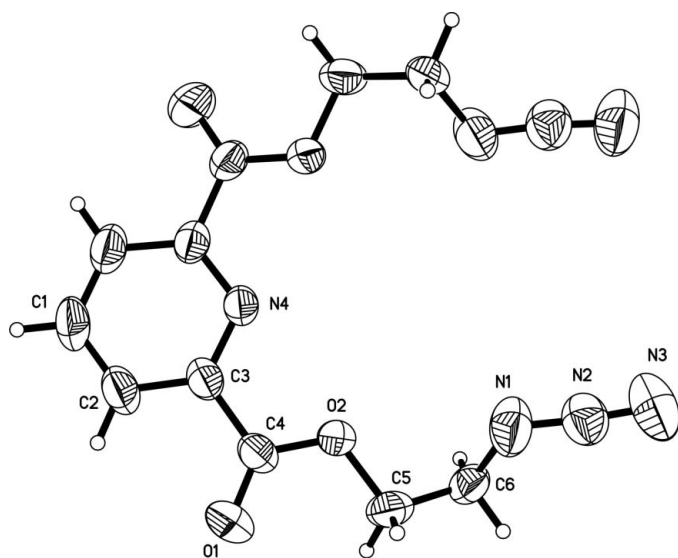


Figure 1
The structure of the compound (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

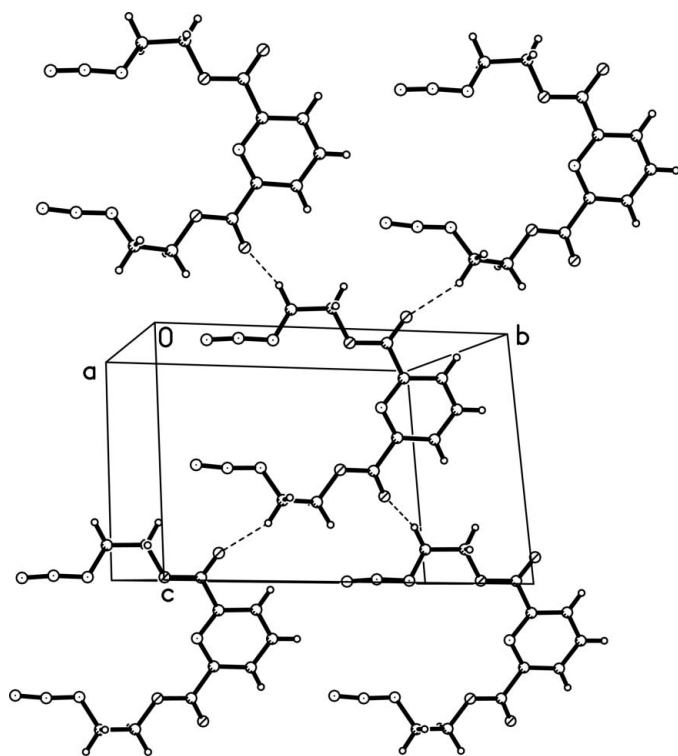


Figure 2
A view down the *a* axis, showing the two-dimensional layer. Hydrogen bonds are indicated by dashed lines.

2-azidoethanol (1.5 g, 13.2 mmol) and triethylamine (1.84 ml, 13.2 mmol) in dry CH_2Cl_2 (15 ml) were added. The mixture was stirred overnight at 323 K and then filtered. Evaporation of the solvent left a crude yellow solid. Chromatographic purification was carried out on a silica-gel column with ethyl acetate–petroleum ether (1:8 *v/v*) as eluants. Colorless single crystals suitable for X-ray crystallographic analysis were grown by slow evaporation of an ethyl acetate solution.

Crystal data

$\text{C}_{11}\text{H}_{11}\text{N}_7\text{O}_4$
 $M_r = 305.27$
 Monoclinic, $C2/c$
 $a = 14.407$ (3) Å
 $b = 11.854$ (3) Å
 $c = 8.942$ (2) Å
 $\beta = 111.142$ (3)°
 $V = 1424.3$ (6) Å³
 $Z = 4$

$D_x = 1.424$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 1833 reflections
 $\theta = 2.3$ – 25.6 °
 $\mu = 0.11$ mm⁻¹
 $T = 293$ (2) K
 Needle, colorless
 $0.36 \times 0.14 \times 0.11$ mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.961$, $T_{\max} = 0.988$
 4299 measured reflections

1416 independent reflections
 1233 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.017$
 $\theta_{\max} = 26.1$ °
 $h = -16 \rightarrow 17$
 $k = -14 \rightarrow 10$
 $l = -11 \rightarrow 10$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.137$
 $S = 1.06$
 1416 reflections
 101 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0749P)^2 + 0.6085P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.20$ e Å⁻³
 $\Delta\rho_{\min} = -0.24$ e Å⁻³

Table 1

Selected bond lengths (Å).

O1–C4	1.204 (2)	N1–N2	1.215 (2)
O2–C4	1.321 (2)	N1–C6	1.465 (2)
O2–C5	1.453 (2)	N2–N3	1.125 (2)

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
C6–H6A···O1 ⁱ	0.97	2.44	3.280 (3)	145

Symmetry code: (i) $-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{3}{2}$.

All H atoms were located in difference Fourier maps and constrained to ride on their parent atoms, with C–H distances in the range 0.93–0.97 Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 1997); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL, PARST (Nardelli, 1995) and PLATON (Spek, 2003).

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