Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis(2-azidoethyl) terephthalate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.045
wR factor = 0.123
Data-to-parameter ratio = 13.7

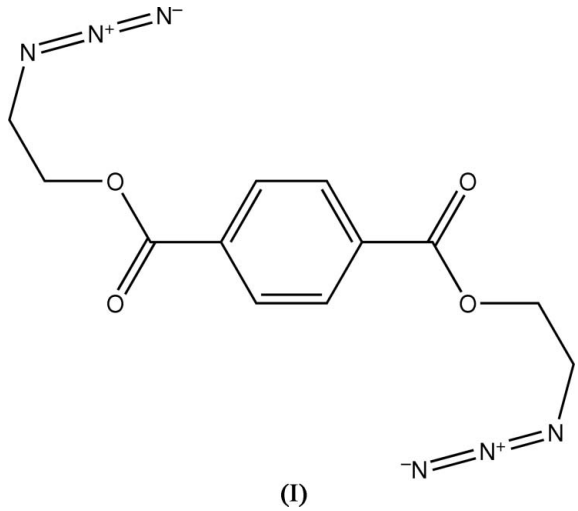
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 13 January 2006
Accepted 13 February 2006

The molecule of the title compound, $C_{12}H_{12}N_6O_4$, is arranged around an inversion center located at the mid-point of the benzene ring. In the crystal structure, molecules are linked into ribbons along the *a* axis by weak C—H···O hydrogen bonds.

Comment

We have reported the structure of tris(2-azidoethyl) benzene-1,3,5-tricarboxylate, (II) (Zhang *et al.*, 2006). As part of our ongoing studies of triazoles, the title compound, (I), has been synthesized as an intermediate product.



(I)

The asymmetric unit of (I) contains one half-molecule, the other half being generated by an inversion centre. All bond lengths and angles are comparable to those in (II). The molecule is essentially planar, excluding the two azide groups which point up and down with repsect to the mean plane. In the crystal structure, molecules are linked into ribbons along the *a* axis (Fig. 2) by weak C—H···O hydrogen bonds (Table 1).

Experimental

A mixture of terephthalic acid (1.0 g, 6.0 mmol) and thionyl chloride (3 ml) was refluxed for 3 h. To this solution were added 2-azidoethanol (1.5 g, 13.2 mmol) and triethylamine (1.84 ml, 13.2 mmol) in dry CH_2Cl_2 (15 ml). The mixture was stirred overnight at 323 K and then filtered. Evaporation of the solvent left a crude yellow solid; this was followed by chromatographic purification on a silica-gel column with ethyl acetate–petroleum ether (1:6 *v*/*v*) as eluants. Colourless single crystals suitable for X-ray crystallographic analysis were grown by slow evaporation of this solution.

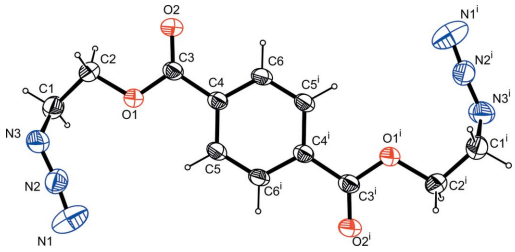


Figure 1
The structure of the compound (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. [Symmetry code: (i) $-x$, $1-y$, $1-z$.]

Crystal data

$C_{12}H_{12}N_6O_4$
$M_r = 304.28$
Triclinic, $P\overline{1}$
$a = 5.2833\ (8)$ Å
$b = 5.7039\ (8)$ Å
$c = 12.0883\ (18)$ Å
$\alpha = 88.052\ (2)^\circ$
$\beta = 85.975\ (2)^\circ$
$\gamma = 81.389\ (2)^\circ$
$V = 359.19\ (9)$ Å^3
$Z = 1$
$D_x = 1.407$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 824 reflections
$\theta = 3.6$–25.9°
$\mu = 0.11$ mm^{-1}
$T = 293\ (2)$ K
Column, colourless
0.26 × 0.15 × 0.12 mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.972$, $T_{max} = 0.987$
2001 measured reflections
1366 independent reflections
1104 reflections with $I > 2\sigma(I)$
$R_{int} = 0.007$
$\theta_{max} = 26.0^\circ$
$h = -6 \rightarrow 6$
$k = -7 \rightarrow 6$
$l = -14 \rightarrow 9$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.123$
$S = 1.04$
1366 reflections
100 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0609P)^2 + 0.0623P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.12$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6A···O2^{i}	0.93	2.57	3.341 (2)	140

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

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 $U_{iso}(H) = 1.2U_{eq}(C)$.

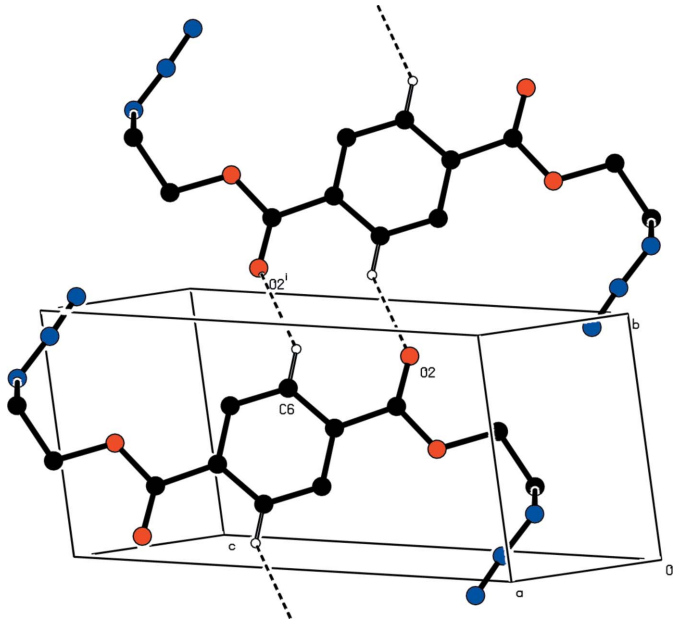


Figure 2
A view showing the C—H···O hydrogen bonds and the formation of ribbons running parallel to the *a* axis. Hydrogen bonds are indicated by dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity. [Symmetry code: (i) $1-x, -y, 1-z$.]

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: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON*.

This project was supported by the Special Project of Qingdao for Generalship of Science and Technology (No. 05–2-JC-80) and the Outstanding Adult-Young Scientific Research Encouraging Foundation of Shandong Province (No. 2005BS04007).

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