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Tris(2-azidoethyl) benzene-1,3,5-tricarboxylate

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In the title molecule, $C_{15}H_{15}N_9O_6$, all bond lengths and angles are within normal ranges. In the crystal structure, molecules are linked into centrosymmetric dimers by weak C—H···O hydrogen bonds.

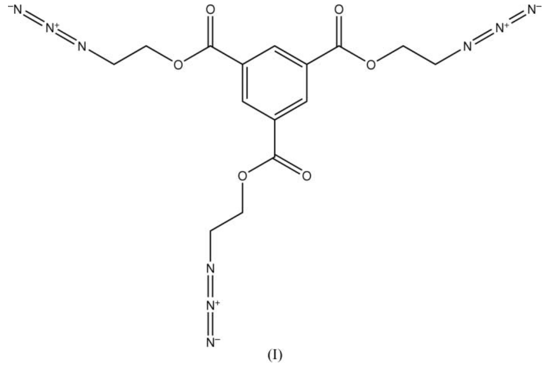
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.065
wR factor = 0.191
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.

Comment

Azides are used to synthesize triazole or tetrazole derivatives, which frequently exhibit significant biological activity and, as a consequence, have found a wide variety of applications as active heterocycles in pharmacy and agriculture (Ostrovskii & Koren, 2000; Nomiya *et al.*, 2000). As part of our studies of triazoles, the title compound, (I), was synthesized by the reaction of 1,3,5-tricarboxylbenzoic acid and 2-azidoethanol.



(I)

All bond lengths and angles in (I) are within normal ranges (Allen *et al.*, 1987). The dihedral angles between the benzene ring and the three azide groups (N1–N3/C9, N4–N6/C15 and

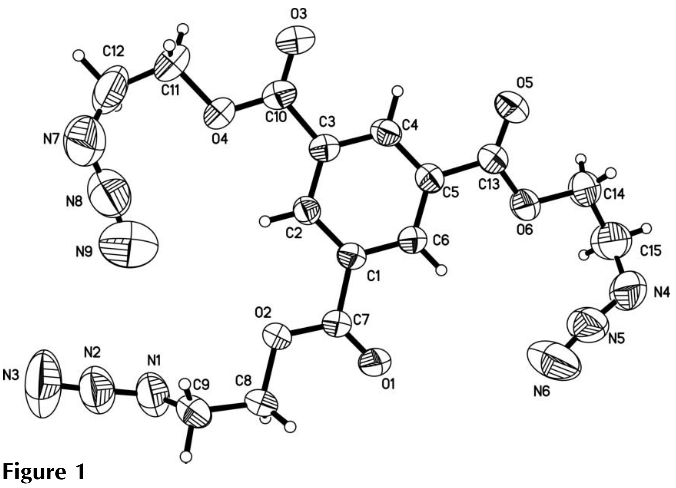


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids.

N7–N9/C12) are 86.8 (3), 47.3 (4) and 41.5 (4)°, respectively. In the crystal structure, molecules are linked into dimers (Fig. 2) by weak C—H···O hydrogen bonds (Table 2).

Experimental

A mixture of benzene-1,3,5-tricarboxylic acid (1.0 g, 5 mmol) and thionyl chloride (3 ml) was refluxed for 3 h. To this solution were added 2-azidoethanol (1.37 g, 15 mmol) and triethylamine (2.17 ml, 16 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 using an ethyl acetate–petroleum ether (1:8 *v*/*v*) mixture as eluant. Colourless single crystals suitable for X-ray crystallographic analysis were grown by slow evaporation of an ethyl acetate solution of (I).

Crystal data

$C_{15}H_{15}N_9O_6$
$M_r = 417.36$
Triclinic, $P\overline{1}$
$a = 6.7391$ (9) Å
$b = 10.6986$ (14) Å
$c = 14.5075$ (19) Å
$\alpha = 100.108$ (2)°
$\beta = 98.961$ (2)°
$\gamma = 106.181$ (2)°
$V = 965.5$ (2) Å^3
$Z = 2$
$D_x = 1.436$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1412 reflections
$\theta = 2.2$–24.5°
$\mu = 0.12$ mm^{-1}
$T = 293$ (2) K
Block, colourless
0.21 × 0.11 × 0.09 mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.976$, $T_{\max} = 0.990$
5138 measured reflections
3718 independent reflections
2672 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.015$
$\theta_{\max} = 26.0°$
$h = -8 \rightarrow 8$
$k = -8 \rightarrow 13$
$l = -17 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.065$
$wR(F^2) = 0.191$
$S = 1.03$
3718 reflections
271 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0947P)^2 + 0.3141P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.46$ e Å^{-3}
$\Delta\rho_{\min} = -0.24$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C7	1.205 (3)	O4—C11	1.448 (3)
O2—C7	1.327 (3)	O5—C13	1.202 (3)
O2—C8	1.458 (3)	O6—C13	1.330 (3)
O3—C10	1.204 (3)	O6—C14	1.450 (3)
O4—C10	1.329 (3)		
N3—N2—N1	174.7 (4)	N9—N8—N7	172.7 (5)
N6—N5—N4	173.6 (5)		

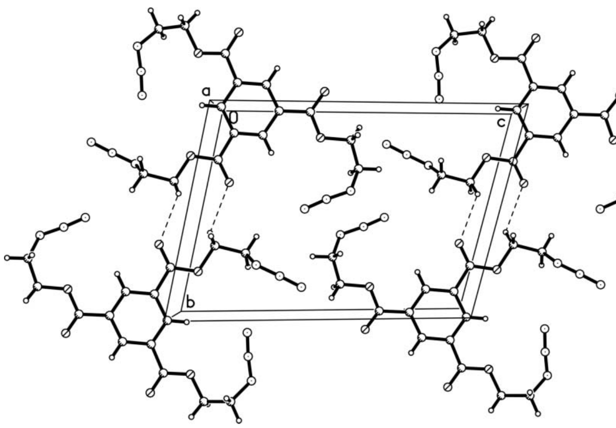


Figure 2
Unit-cell projection along the *a* axis. Hydrogen bonds are indicated by dashed lines.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8*A*···O1$^{\rm i}$	0.97	2.58	3.175 (3)	120

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

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 $U_{\rm iso}$(H) = 1.2 $U_{\rm eq}$(C) for all H atoms.

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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