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3,3′-(4-Amino-4*H*-1,2,4-triazole-3,5-diyl)dibenzoic acid

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.058
wR factor = 0.167
Data-to-parameter ratio = 11.8

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

The title compound, $C_{16}H_{12}N_4O_4$, has a non-planar molecular structure. An intermolecular hydrogen-bonding network helps to stabilize the crystal structure.

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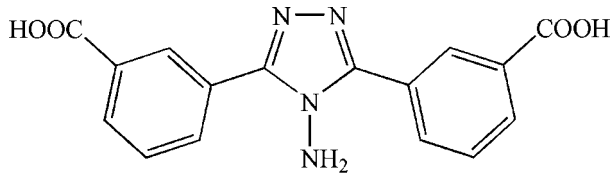
Comment

Dicarboxylates have been used in preparing coordination polymers (Yaghi *et al.*, 1995). As part of our ongoing investigation of polymeric complexes (Dong *et al.*, 2005), we recently prepared the title dicarboxylic acid, (I), involving a 1,2,4-triazole group, and have determined its crystal structure.



(I)

The molecular structure of (I) is shown in Fig. 1. The three aromatic rings in (I) are twisted relative to each other: the dihedral angles between the triazole and C2-benzene rings and between the triazole and C10-benzene rings are 16.91 (17) and 46.68 (16)°, respectively.

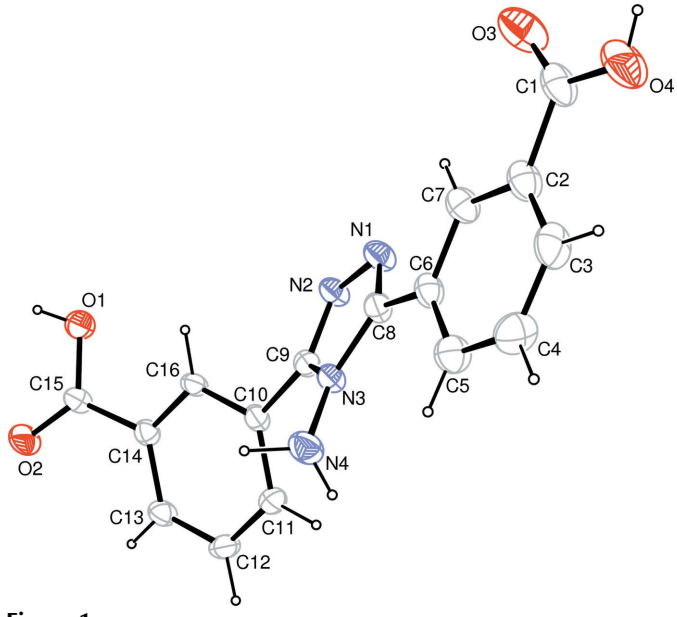


Figure 1
The molecular structure of (I), with 30% probability displacement ellipsoids (arbitrary spheres for H atoms).

An intermolecular hydrogen-bonding network helps to stabilize the crystal structure.

Experimental

3-Cyanobenzoic acid (2.2 g, 15 mmol), hydrazine sulfate (1.95 g, 15 mmol), 80% hydrazine hydrate (2.8 g, 45 mmol) and glycol (8 ml) were mixed under a nitrogen atmosphere and heated at 403 K for 3 h. The mixture was then cooled to room temperature and poured into water (50 ml); 35% HCl solution (2.5 ml) was then added to yield a white precipitate (2.0 g, 90%). The precipitate (16.2 mg, 0.05 mol) and water (2 ml) were sealed in a 6 ml glass tube and heated at 453 K for 3 d, then cooled to room temperature to obtain single crystals of (I).

Crystal data

$C_{16}H_{12}N_4O_4$
M_r = 324.30
Monoclinic, $C2/c$
a = 36.92 (1) Å
b = 7.466 (2) Å
c = 10.868 (3) Å
β = 104.038 (4)°
V = 2906.1 (13) Å^3
Z = 8
D_x = 1.482 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 298 (2) K
Block, colourless
0.32 × 0.12 × 0.08 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
6351 measured reflections
2572 independent reflections
1726 reflections with $I > 2\sigma(I)$
R_{int} = 0.046
θ_{max} = 25.2°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.167
S = 1.06
2572 reflections
218 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0808P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N2^{i}	0.82	1.84	2.635 (3)	164
O4—H4C···O3^{ii}	0.80	1.84	2.639 (4)	174
N4—H4A···N1^{iii}	0.96	2.27	3.216 (3)	168
N4—H4B···O2^{iv}	0.86	2.42	3.194 (4)	150

Symmetry codes: (i) $x, -y-1, z-\frac{1}{2}$; (ii) $-x+1, -y, -z+3$; (iii) $x, -y, z-\frac{1}{2}$; (iv) $x, -y, z+\frac{1}{2}$.

H atoms on O and N atoms were located in a difference Fourier map and refined as riding in their as-found relative positions, with U_{iso}(H) = 1.5U_{eq}(O,N). Other H atoms were placed in calculated positions, with C—H = 0.93 Å, and refined in riding mode, with U_{iso}(H) = 1.2U_{eq}(C).

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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