

2,2'-(Propane-1,3-diimino)dibenzoic acid

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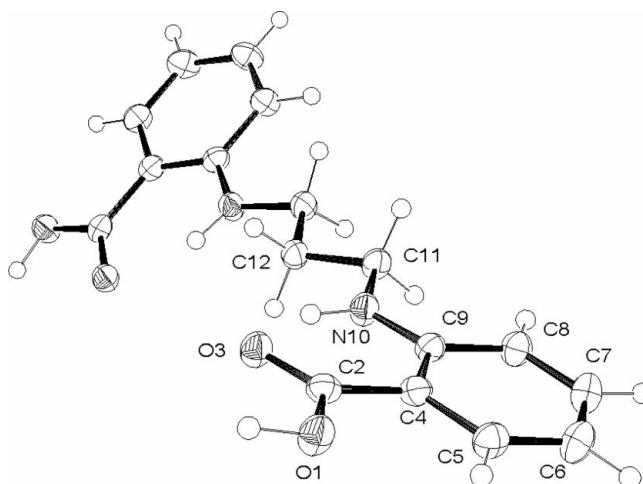
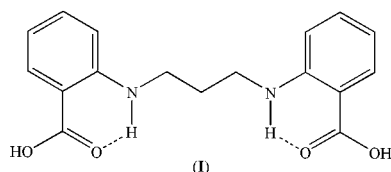
Key indicators

Single-crystal X-ray study
 $T = 100$ K
Mean $\sigma(C-C) = 0.002$ Å
 R factor = 0.042
 wR factor = 0.118
Data-to-parameter ratio = 14.3For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The crystal structure of the title compound, $C_{17}H_{18}N_2O_4$, is presented. The molecule sits on a crystallographic twofold rotation axis. It contains $O \cdots H-N$ intramolecular hydrogen bonds typical of *ortho*-benzoic amino acids; $O \cdots H-O$ intermolecular hydrogen bonds connect the molecules into infinite chains.

Comment

The title compound, (I), was synthesized as part of our effort to produce new ligands for the uranyl cation (UO_2^{2+}) and for structural studies with Cr^{III} . Similar ligands have been shown to adopt an equatorial binding mode when bound to Cu^{II} in the presence of an axial coordinating ligand (Berger & Telford, 2002). In this coordination complex, the parallel geometry of amine N–H bonds enforces dimerization by molecular self-recognition, forming strong intermolecular hydrogen-bonding interactions. The structure determination of (I) was conducted in order to obtain size and stereochemical information. The results of the single-crystal X-ray study are consistent with the stoichiometry and connectivity of the proposed formulation (Fig. 1).

**Figure 1**

The molecular structure of (I), showing displacement ellipsoids at the 50% probability level. The numbering scheme of the asymmetric unit is shown.

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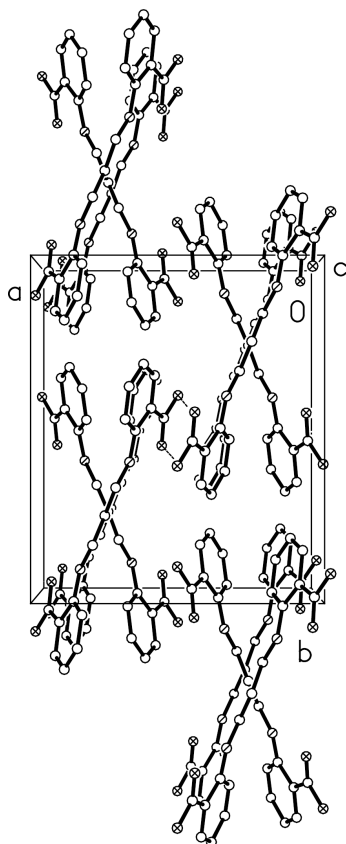


Figure 2
Packing diagram for (I). Intermolecular hydrogen bonding through the carboxylate groups is indicated by dashed lines on the central pair of molecules. Hydrogen atoms have been omitted for clarity.

A planar geometry for (I) would place it in a C_{2v} or C_{2h} point group. In the solid state, hydrogen-bonding interactions enforce a geometry giving rise to C_2 symmetry; the orientation of the two aromatic rings is *gauche*, the angle between the planes being $44.5 (5)^\circ$. The molecule sits on a crystallographic twofold rotation axis, and thus only half of the atoms in the molecule are unique. The $N-C_{\text{arom}}$ bond [$C9-N10 = 1.366 (2) \text{ \AA}$] is shorter than the $N-C_{\text{aliph}}$ bond [$C11-N10 = 1.450 (2) \text{ \AA}$; Table 1]. The torsion angle between the aromatic ring and the *N*-methylene groups ($C4-C9-N10-C11$) is $175.0 (1)^\circ$ and that of the $C2-C4-C9-N10$ fragment is $-4.9 (2)^\circ$. The molecules in the crystal structure are interconnected by $O1-H1 \cdots O3$ hydrogen bonding (Table 2), as shown in Fig. 2. This compound also contains intramolecular hydrogen bonds ($N10-H10 \cdots O3$) between amine and carboxyl groups.

Experimental

The title compound was prepared according to a slight modification of the previously reported method (Sandhu & Verma, 1986). Anthranilic acid (54.8 g, 0.4 mol) was added to 150 ml of ethanol in a 500 ml round-bottom flask. To this solution, 1,3-dibromoethane (20.8 ml, 0.24 mol) was added and the mixture was allowed to reflux overnight. A yellow crystalline compound, including X-ray diffraction quality crystals, was obtained after cooling the reaction mixture or after simple recrystallization. Slower crystallization conditions (*viz.*

cooling with a heat sink) led to twinned and clustered crystals. The compound was collected by filtration, washed with hot acetone ($2 \times 20 \text{ ml}$) and glacial acetic acid ($2 \times 20 \text{ ml}$), and then dried *in vacuo*. Yield 28%, m.p. 472.3–473.7 K (differs from previous report, 493–494 K). $^1\text{H NMR}$ (d_6 -DMSO): δ 12.82 (*br, s*, 2H), 8.37 (*dd*, $J_{\text{HH}} = 7.9$ and 1.7 Hz , 1H), 7.92 (*ddd*, $J_{\text{HH}} = 8.6, 7.0$ and 1.7 Hz , 1H), 7.32 (*d*, $J_{\text{HH}} = 8.6 \text{ Hz}$, 1H), 7.13 (*ddd*, $J_{\text{HH}} = 7.9, 7.0$ and 1.0 Hz , 1H), 3.87 (*m*, 4H), 3.08 (*m*, 2H).

Crystal data

$C_{17}H_{18}N_2O_4$
 $M_r = 314.33$
Orthorhombic, $Pccn$
 $a = 11.9318 (18) \text{ \AA}$
 $b = 14.152 (2) \text{ \AA}$
 $c = 9.0323 (14) \text{ \AA}$
 $V = 1525.2 (4) \text{ \AA}^3$
 $Z = 4$
 $D_x = 1.369 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation
Cell parameters from 4874 reflections
 $\theta = 2.2\text{--}26.0^\circ$
 $\mu = 0.10 \text{ mm}^{-1}$
 $T = 100 (2) \text{ K}$
Block, yellow
 $0.48 \times 0.17 \times 0.16 \text{ mm}$

Data collection

Bruker SMART APEX CCD area-detector diffractometer
 ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2000)
 $T_{\text{min}} = 0.954, T_{\text{max}} = 0.984$
8887 measured reflections

1504 independent reflections
1382 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$
 $\theta_{\text{max}} = 26.0^\circ$
 $h = -14 \rightarrow 13$
 $k = -17 \rightarrow 16$
 $l = -11 \rightarrow 11$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.118$
 $S = 1.02$
1504 reflections
105 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.071P)^2 + 0.67P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.35 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.32 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters ($\text{\AA}, ^\circ$).

O1—C2	1.322 (2)	C4—C9	1.416 (2)
C2—O3	1.240 (2)	C9—N10	1.366 (2)
C2—C4	1.464 (2)	N10—C11	1.450 (2)
O3—C2—O1	120.9 (1)	N10—C9—C4	121.5 (1)
O3—C2—C4	123.8 (1)	N10—C9—C8	120.7 (1)
O1—C2—C4	115.2 (1)	C4—C9—C8	117.8 (1)
C5—C4—C9	119.6 (1)	C9—N10—C11	124.6 (1)
C5—C4—C2	118.6 (1)	N10—C11—C12	109.3 (1)
C9—C4—C2	121.8 (1)	C11 ⁱ —C12—C11	112.3 (1)
O3—C2—C4—C9	1.3 (2)	C4—C9—N10—C11	175.0 (1)
O1—C2—C4—C9	-178.5 (1)	N10—C11—C12—C11 ⁱ	175.0 (1)
C2—C4—C9—N10	-4.9 (2)		

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

Table 2

Hydrogen-bonding geometry ($\text{\AA}, ^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O1—H1 \cdots O3 ⁱⁱ	0.95	1.69	2.626 (1)	166
N10—H10 \cdots O3	0.88	2.01	2.682 (1)	132

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

H-atom positions were initially determined by geometry and refined by a riding model. H-atom displacement parameters were set to 1.2 times the displacement parameters of the bonded atoms.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINTE* (Bruker, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1994); software used to prepare material for publication: *SHELXTL*.

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