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5,5′-Oxybis[2-(phthalimido)acetic acid] *N*,*N*-dimethylformamide disolvate

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Key indicators

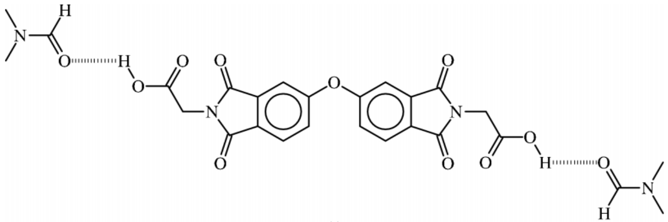
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
Disorder in solvent or counterion
R factor = 0.060
wR factor = 0.196
Data-to-parameter ratio = 15.0

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

The two *N*-pthaloylglycine moieties of the title compound, $C_{20}H_{12}N_2O_9 \cdot 2C_3H_7NO$, are connected through an ether linkage, which apparently has sp^2 character [O—C = 1.376 (3) and 1.392 (3) Å; C—O—C = 120.3 (2)°]. The planar *N*-phthaloyl units are inclined to each other by 65.2 (1)°.

Comment

Amino acids readily participate in condensation reactions with phthalic anhydride to yield *N*-phthaloylaminoacids, crystalline compounds that can be identified by their melting points. *N*-Phthaloylglycine is synthesized by merely melting glycine with phthalic anhydride (Furniss *et al.*, 1978) and the yellow 3-nitro derivative can be obtained in a similar way (Ng *et al.*, 1991). These protected amino acids have been condensed with triorganotin hydroxide/hemioxide to afford biologically active triorganotin *N*-phthaloylaminoacetates (Lo *et al.*, 1997; Ng & Kumar Das, 1997; Ng *et al.*, 1990, 1994; Ovsetsina *et al.*, 1993).



(I)

The synthesis of the title compound, (I), required the use of DMF as solvent, which was incorporated into the crystal structure. The two *N*-phthaloylglcinate moieties are linked by the ether atom O5, which displays some sp^2 character [O5—C7 = 1.376 (3) Å, O5—C11 = 1.392 (3) Å and C7—O5—C1 = 120.3 (2)°] (Fig. 1). The *N*-phthaloyl units are planar, and are inclined to each other by 65.2 (1)°. Unlike a large number of carboxylic acids that are usually hydrogen bonded to each other in the crystal structure, in the title compound, each of the two carboxylic acid groups interacts with a DMF molecule through a short hydrogen bond [O1···O10 = 2.607 (4) Å and O9···O11 = 2.57 (1) Å (the latter distance represents an average over distances involving two disordered components)].

Experimental

The title compound was prepared by the condensation of 4,4′-oxydiphthalic anhydride (3.1 g, 10 mmol), a commercially available

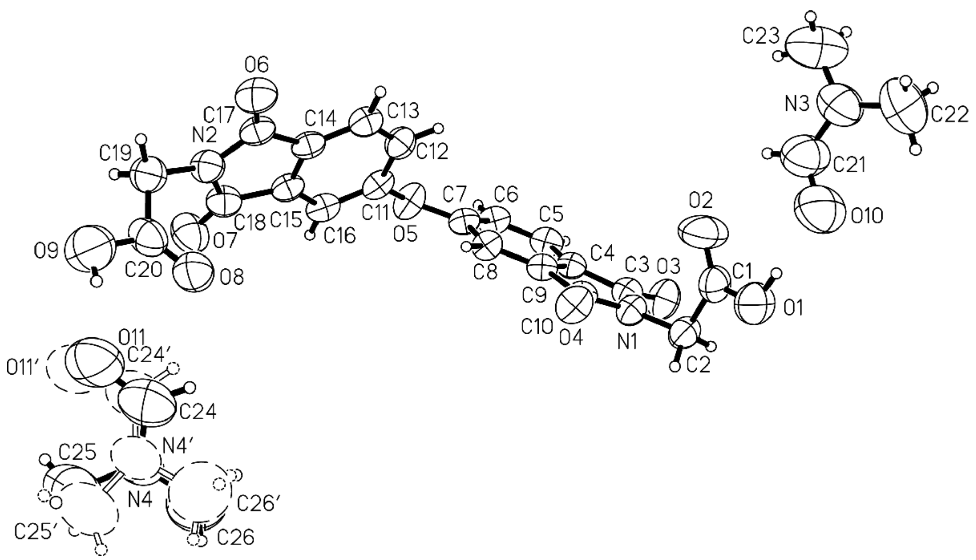


Figure 1

*ORTEP*II (Johnson, 1976) plot of (I). Displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as spheres of arbitrary radii. The minor disordered component is indicated by primed atom labels and broken-line ellipsoids.

chemical, and glycine (1.5 g, 20 mmol) in DMF (30 ml). The solution was heated under reflux for 5 h. A solid product that separated on cooling was collected by filtration in 70% yield. The pure product was obtained by recystallization from a DMF/water (20 ml/5 ml) mixture.

Crystal data

$C_{20}H_{12}N_2O_9{\cdot}2C_3H_7NO$
$M_r = 570.51$
Triclinic, $P\overline{1}$
$a = 8.357$ (1) Å
$b = 11.122$ (2) Å
$c = 15.044$ (2) Å
$\alpha = 93.053$ (2)°
$\beta = 96.081$ (2)°
$\gamma = 90.891$ (2)°
$V = 1388.2$ (3) Å^3

$Z = 2$
$D_x = 1.365$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 805 reflections
$\theta = 2.4$–23.6°
$\mu = 0.11$ mm^{-1}
$T = 298$ (2) K
Block, colorless
0.50 × 0.39 × 0.14 mm

Data collection

Bruker SMART area-detector diffractometer
φ and ω scans
Absorption correction: none
11580 measured reflections
5937 independent reflections

3216 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.028$
$\theta_{\rm max} = 27.1°$
$h = -10 \rightarrow 10$
$k = -14 \rightarrow 14$
$l = -19 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.060$
$wR(F^2) = 0.196$
$S = 1.00$
5937 reflections
397 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.1001P)^2 + 0.3122P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.45$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.26$ e Å^{-3}

Table 1

Selected geometric parameters (Å, °).

O1—C1	1.278 (4)	C3—C4	1.471 (4)
O2—C1	1.200 (4)	C4—C5	1.378 (4)
O3—C3	1.209 (3)	C4—C9	1.389 (3)
O4—C10	1.210 (3)	C5—C6	1.374 (4)
O5—C7	1.376 (3)	C6—C7	1.385 (4)
O5—C11	1.392 (3)	C7—C8	1.394 (4)
O6—C17	1.204 (3)	C8—C9	1.369 (4)
O7—C18	1.199 (3)	C9—C10	1.485 (4)
O8—C20	1.190 (4)	C11—C16	1.375 (4)
O9—C20	1.294 (4)	C11—C12	1.383 (4)
N1—C2	1.442 (4)	C12—C13	1.376 (4)
N1—C3	1.390 (3)	C13—C14	1.369 (4)
N1—C10	1.383 (3)	C14—C15	1.384 (4)
N2—C17	1.390 (4)	C14—C17	1.480 (4)
N2—C18	1.398 (4)	C15—C16	1.375 (4)
N2—C19	1.442 (4)	C15—C18	1.483 (4)
C1—C2	1.499 (4)	C19—C20	1.514 (5)
C7—O5—C11	120.3 (2)	O4—C10—N1	124.5 (2)
C10—N1—C2	121.6 (2)	O4—C10—C9	129.7 (3)
C10—N1—C3	112.2 (2)	N1—C10—C9	105.8 (2)
C2—N1—C3	125.4 (2)	C12—C11—O5	120.7 (3)
C17—N2—C18	112.0 (2)	C16—C11—O5	116.5 (2)
C17—N2—C19	124.7 (2)	C12—C11—C16	122.6 (2)
C18—N2—C19	122.5 (3)	C11—C12—C13	120.2 (3)
O1—C1—O2	124.4 (3)	C12—C13—C14	117.8 (3)
O1—C1—C2	113.9 (3)	C13—C14—C15	121.4 (2)
O2—C1—C2	121.7 (3)	C13—C14—C17	130.3 (2)
N1—C2—C1	110.1 (2)	C15—C14—C17	108.3 (2)
O3—C3—N1	124.3 (3)	C14—C15—C16	121.6 (3)
O3—C3—C4	129.6 (3)	C14—C15—C18	108.2 (2)
N1—C3—C4	106.0 (2)	C16—C15—C18	130.2 (3)
C3—C4—C5	131.5 (2)	C11—C16—C15	116.4 (2)
C3—C4—C9	108.2 (2)	O6—C17—N2	124.6 (3)
C5—C4—C9	120.3 (2)	O6—C17—C14	129.5 (3)
C4—C5—C6	118.4 (2)	N2—C17—C14	105.9 (2)
C5—C6—C7	120.6 (2)	O7—C18—N2	124.4 (3)
O5—C7—C6	115.2 (2)	O7—C18—C15	130.0 (3)
O5—C7—C8	122.7 (2)	N2—C18—C15	105.6 (2)
C6—C7—C8	121.9 (2)	N2—C19—C20	110.1 (3)
C7—C8—C9	116.3 (2)	O8—C20—O9	126.1 (4)
C4—C9—C8	122.5 (2)	O8—C20—C19	123.8 (3)
C4—C9—C10	107.8 (2)	O9—C20—C19	110.1 (3)
C8—C9—C10	129.7 (2)	O10—C21—N3	124.7 (5)

One of the two DMF molecules is disordered over two positions; since the occupancies could not be refined, the disorder was treated as a 50/50 disorder. The displacement parameters of the unprimed and primed atoms (Fig. 1) were kept equal; additionally, pairs of bonds were restrained to be approximately equal. The displacement parameter of the carboxyl O9 atom that is engaged in hydrogen bonding with this DMF (O11, N4, C24, C25, C26) was restrained to be approximately isotropic. The displacement parameters of the atoms of the disordered DMF were similarly restrained.

The H atoms were placed at calculated positions (C—H = 0.93 Å for the aromatic H atoms, C—H = 0.97 Å for the methylene H atoms and 0.96 Å for the methyl H atoms) and refined in the riding model approximation. The two carboxylic acid H atoms were located and refined with the O—H bonds restrained to 0.85 (1) Å. The displacement parameters of the H atoms were set at 1.2 times $U_{\rm eq}$ of the parent atoms, except for the methyl H atoms for which the ratio was 1.5.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1999); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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