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N,*N*′-Dibenzyl-2,2′-ethylenedioxydibenzamide

Hui-Ling Wen, Qing-Hua Huang and Yong-Hong Wen*

College of Chemistry and Molecular Engineering, Qingdao University of Science and Technology, 266042 Qingdao, Shandong, People's Republic of China
Correspondence e-mail: qustchemistry@126.com

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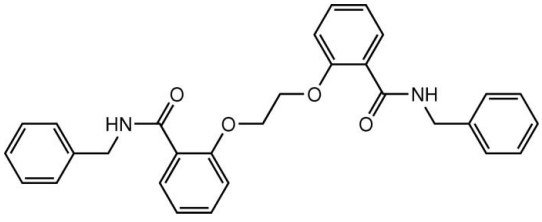
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.069; wR factor = 0.207; data-to-parameter ratio = 14.4.

The asymmetric unit of the title compound, $C_{30}H_{28}N_2O_4$, contains one-half of the centrosymmetric molecule. All bond lengths and angles show normal values. Weak intermolecular N—H···O hydrogen bonds link molecules related by translation along the *a* axis into chains.

Related literature

The structure of 1,2-ethylenedioxy-bis(*N*-pyridin-2-ylmethylbenzamide) was reported by Wen & Zhang (2007). For general background, see: West *et al.* (1992); Bunzli *et al.* (1984); Wen *et al.* (2002); Allen *et al.* (1987).



Experimental

Crystal data

$C_{30}H_{28}N_2O_4$
M_r = 480.54
Monoclinic, $P2_1/c$
a = 5.0620 (13) Å
b = 16.401 (4) Å
c = 15.056 (4) Å
β = 96.522 (5)°
V = 1241.9 (6) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 (2) K
0.42 × 0.11 × 0.05 mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.989, T_{max} = 0.996
6638 measured reflections
2341 independent reflections
1425 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.069
$wR(F^2)$ = 0.207
S = 1.12
2341 reflections
163 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	0.86	2.19	2.895 (3)	139

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2330).

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supplementary materials

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N,*N*'-Dibenzyl-2,2'-ethylenedioxydibenzamide

H.-L. Wen, Q.-H. Huang and Y.-H. Wen

Comment

The acyclic polyether compounds have good complexing ability and high selectivity to metal ions. Of which diamide-type acyclic polyethers have been used successfully as the active materials for ion-selective electrodes and extractants for metal ions (West *et al.*, 1992; Bunzli *et al.*, 1984; Wen *et al.*, 2002). Recently, in our ongoing studies of structures and properties of the rare earth complexes with diamide-type acyclic polyethers (Wen & Zhang, 2007), a new flexible acyclic polyether compound 1,2-ethylenedioxy-bis(*N*-phenylmethyl-benzamide) (I), was synthesized. Herein we report the synthesis and structural characterization of (I).

The asymmetric part of the title compound contains a half of the centrosymmetric molecule, with the midpoint of the C15—C15^{ii} bond [symmetry code: (ii): $1-x, 1-y, -z$] located on an invesrion center (Fig. 1). All bond lengths and angles in (I) show normal values (Allen *et al.*, 1987).

In the crystal structure, molecules are linked into chains along *a* axis by weak N—H···O intermolecular hydrogen bonds (Table 1 and Fig. 2).

Experimental

1,2-Dichloroethane (0.99 g, 0.01 mol) was added dropwise to a 50 ml of DMF solution containing 2-hydroxy-*N*-phenylmethyl-benzamide (4.54 g, 0.02 mol), K_2CO_3 (3 g, 0.02 mol) and KI (0.5 g), and the mixture was stirred at 328 K for 48 h. After cooling to room temperature, the mixture was filtered. DMF was removed from the filtrate under reduced pressure, and the reside was washed by column chromatography (silica gel, C_2H_5OH:$CH_3CO_2C_2H_5$ = 1:4) resulting in a colourless solid. Colourless single crystals of (I) suitable for X-ray diffraction study were obtained by slow evaporation of an ethanol–DMF (1:1 *v*/*v*) solution over a period of 25 d.

Refinement

All H atoms were located in difference Fourier maps, placed in idealized positions (C—H 0.93–0.98 Å, N—H 0.86 Å) and refined as riding, with U_{iso}(H) = 1.2 U_{eq}(C, N).

Figures

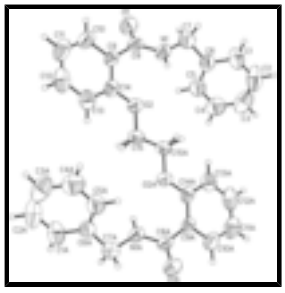

Fig. 1. The molecular structure of (I) showing 50% probability displacement ellipsoids and the atom numbering scheme [symmetry code: (A) $1-x$, $1-y$, $1-z$].

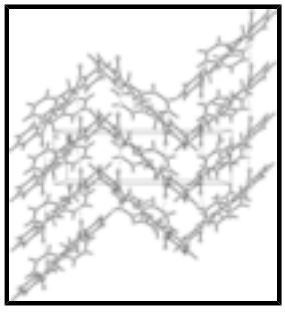

Fig. 2. A packing diagram of (I), viewed down the *c* axis. Hydrogen bonds are indicated by dashed lines.

_N_,_N_'-Dibenzyl-2,2'-ethylenedioxydibenzamide

Crystal data

$C_{30}H_{28}N_2O_4$	F_{000} = 508
M_r = 480.54	D_x = 1.285 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 5.0620 (13) Å	Cell parameters from 934 reflections
b = 16.401 (4) Å	θ = 2.5–21.0º
c = 15.056 (4) Å	μ = 0.09 mm^{-1}
β = 96.522 (5)º	T = 293 (2) K
V = 1241.9 (6) Å^3	Block, colourless
Z = 2	0.42 × 0.11 × 0.05 mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer	2341 independent reflections
Radiation source: fine-focus sealed tube	1425 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.035
Detector resolution: 8.33 pixels mm^{-1}	θ_{max} = 25.7º
T = 293(2) K	θ_{min} = 1.8º
ω scans	h = −6→5
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	k = −19→16
T_{min} = 0.989, T_{max} = 0.996	l = −18→17

6638 measured reflections

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.069$	H-atom parameters constrained
$wR(F^2) = 0.207$	$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.12$	$(\Delta/\sigma)_{max} < 0.001$
2341 reflections	$\Delta\rho_{max} = 0.46$ e Å^{-3}
163 parameters	$\Delta\rho_{min} = -0.33$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O2	0.2700 (4)	0.43132 (12)	0.04071 (12)	0.0539 (6)
C14	0.1064 (5)	0.36654 (17)	0.01572 (19)	0.0478 (7)
C9	−0.0472 (5)	0.33705 (16)	0.08071 (19)	0.0465 (7)
C8	−0.0383 (6)	0.36742 (17)	0.1743 (2)	0.0490 (7)
N1	0.1948 (5)	0.39077 (16)	0.21573 (16)	0.0590 (7)
H1A	0.3314	0.3884	0.1867	0.071*
O1	−0.2399 (4)	0.36622 (16)	0.21280 (15)	0.0808 (8)
C15	0.4239 (5)	0.46574 (17)	−0.02322 (18)	0.0511 (8)
H15B	0.4287	0.4489	−0.0820	0.077*
H15C	0.3052	0.4862	−0.0725	0.077*
C6	0.3986 (6)	0.4961 (2)	0.3189 (2)	0.0564 (8)
C10	−0.2248 (6)	0.27390 (19)	0.0563 (2)	0.0642 (9)
H10A	−0.3315	0.2546	0.0981	0.077*
C13	0.0843 (6)	0.3322 (2)	−0.0687 (2)	0.0622 (9)
H13A	0.1851	0.3524	−0.1118	0.075*
C11	−0.2482 (8)	0.2393 (2)	−0.0265 (3)	0.0785 (11)
H11A	−0.3693	0.1974	−0.0409	0.094*

C12	−0.0899 (7)	0.2673 (2)	−0.0889 (3)	0.0752 (11)
H12A	−0.0996	0.2426	−0.1448	0.090*
C5	0.3608 (7)	0.5599 (2)	0.2607 (2)	0.0712 (10)
H5A	0.2302	0.5562	0.2121	0.085*
C4	0.5126 (9)	0.6296 (2)	0.2724 (3)	0.0817 (11)
H4A	0.4830	0.6725	0.2321	0.098*
C7	0.2325 (7)	0.4201 (2)	0.3075 (2)	0.0742 (10)
H7A	0.3172	0.3776	0.3455	0.089*
H7B	0.0599	0.4309	0.3271	0.089*
C3	0.7041 (8)	0.6360 (3)	0.3422 (3)	0.0908 (13)
H3A	0.8067	0.6830	0.3497	0.109*
C2	0.7467 (9)	0.5735 (3)	0.4013 (3)	0.1026 (15)
H2A	0.8778	0.5778	0.4496	0.123*
C1	0.5938 (7)	0.5033 (2)	0.3893 (2)	0.0802 (11)
H1B	0.6244	0.4605	0.4296	0.096*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0512 (12)	0.0616 (13)	0.0498 (12)	−0.0147 (10)	0.0101 (9)	0.0019 (10)
C14	0.0360 (16)	0.0464 (17)	0.0594 (18)	0.0058 (13)	−0.0022 (13)	0.0022 (14)
C9	0.0309 (14)	0.0425 (16)	0.0642 (18)	0.0008 (12)	−0.0022 (13)	0.0092 (14)
C8	0.0359 (16)	0.0505 (17)	0.0605 (18)	−0.0012 (13)	0.0054 (14)	0.0150 (14)
N1	0.0405 (15)	0.0810 (19)	0.0568 (15)	−0.0144 (13)	0.0107 (12)	−0.0051 (13)
O1	0.0422 (14)	0.125 (2)	0.0772 (16)	−0.0098 (13)	0.0160 (12)	0.0040 (14)
C15	0.0481 (17)	0.063 (2)	0.0423 (15)	−0.0055 (14)	0.0058 (13)	0.0110 (13)
C6	0.0483 (18)	0.069 (2)	0.0524 (17)	−0.0021 (15)	0.0071 (14)	−0.0054 (16)
C10	0.0497 (19)	0.0531 (19)	0.086 (2)	−0.0065 (15)	−0.0081 (17)	0.0112 (18)
C13	0.056 (2)	0.071 (2)	0.059 (2)	0.0062 (17)	0.0014 (15)	−0.0096 (17)
C11	0.072 (3)	0.054 (2)	0.104 (3)	−0.0091 (18)	−0.013 (2)	−0.007 (2)
C12	0.072 (2)	0.068 (2)	0.079 (2)	0.015 (2)	−0.017 (2)	−0.024 (2)
C5	0.066 (2)	0.083 (3)	0.063 (2)	−0.0006 (19)	−0.0006 (17)	−0.0004 (19)
C4	0.093 (3)	0.067 (2)	0.088 (3)	−0.002 (2)	0.018 (2)	−0.005 (2)
C7	0.071 (2)	0.096 (3)	0.056 (2)	−0.019 (2)	0.0081 (17)	0.0016 (18)
C3	0.078 (3)	0.070 (3)	0.126 (4)	−0.006 (2)	0.015 (3)	−0.034 (3)
C2	0.088 (3)	0.096 (3)	0.113 (4)	0.007 (3)	−0.031 (3)	−0.038 (3)
C1	0.084 (3)	0.080 (3)	0.071 (2)	0.006 (2)	−0.019 (2)	−0.0083 (19)

Geometric parameters (Å, °)

O2—C14	1.373 (3)	C10—H10A	0.9300
O2—C15	1.422 (3)	C13—C12	1.394 (5)
C14—C13	1.383 (4)	C13—H13A	0.9300
C14—C9	1.403 (4)	C11—C12	1.380 (5)
C9—C10	1.393 (4)	C11—H11A	0.9300
C9—C8	1.491 (4)	C12—H12A	0.9300
C8—O1	1.229 (3)	C5—C4	1.378 (5)
C8—N1	1.327 (4)	C5—H5A	0.9300
N1—C7	1.455 (4)	C4—C3	1.350 (5)

N1—H1A	0.8600	C4—H4A	0.9300
C15—C15^{i}	1.491 (5)	C7—H7A	0.9700
C15—H15B	0.9300	C7—H7B	0.9700
C15—H15C	0.9599	C3—C2	1.358 (6)
C6—C5	1.364 (5)	C3—H3A	0.9300
C6—C1	1.369 (4)	C2—C1	1.388 (6)
C6—C7	1.503 (5)	C2—H2A	0.9300
C10—C11	1.362 (5)	C1—H1B	0.9300
C14—O2—C15	118.8 (2)	C10—C11—C12	119.2 (3)
O2—C14—C13	123.4 (3)	C10—C11—H11A	120.4
O2—C14—C9	116.1 (2)	C12—C11—H11A	120.4
C13—C14—C9	120.5 (3)	C11—C12—C13	120.5 (3)
C10—C9—C14	117.7 (3)	C11—C12—H12A	119.8
C10—C9—C8	116.6 (3)	C13—C12—H12A	119.8
C14—C9—C8	125.8 (3)	C6—C5—C4	121.2 (3)
O1—C8—N1	121.7 (3)	C6—C5—H5A	119.4
O1—C8—C9	120.1 (3)	C4—C5—H5A	119.4
N1—C8—C9	118.1 (3)	C3—C4—C5	120.3 (4)
C8—N1—C7	123.7 (3)	C3—C4—H4A	119.8
C8—N1—H1A	118.1	C5—C4—H4A	119.8
C7—N1—H1A	118.1	N1—C7—C6	113.2 (3)
O2—C15—C15^{i}	106.0 (3)	N1—C7—H7A	108.9
O2—C15—H15B	127.0	C6—C7—H7A	108.9
C15^{i}—C15—H15B	127.0	N1—C7—H7B	108.9
O2—C15—H15C	108.6	C6—C7—H7B	108.9
C15^{i}—C15—H15C	110.1	H7A—C7—H7B	107.7
H15B—C15—H15C	56.6	C4—C3—C2	119.9 (4)
C5—C6—C1	117.9 (3)	C4—C3—H3A	120.1
C5—C6—C7	121.8 (3)	C2—C3—H3A	120.1
C1—C6—C7	120.3 (3)	C3—C2—C1	119.7 (4)
C11—C10—C9	122.5 (3)	C3—C2—H2A	120.1
C11—C10—H10A	118.7	C1—C2—H2A	120.1
C9—C10—H10A	118.7	C6—C1—C2	121.0 (4)
C14—C13—C12	119.6 (3)	C6—C1—H1B	119.5
C14—C13—H13A	120.2	C2—C1—H1B	119.5
C12—C13—H13A	120.2		
C15—O2—C14—C13	0.5 (4)	C9—C14—C13—C12	−0.7 (4)
C15—O2—C14—C9	−177.9 (2)	C9—C10—C11—C12	0.3 (5)
O2—C14—C9—C10	177.0 (2)	C10—C11—C12—C13	−2.5 (5)
C13—C14—C9—C10	−1.4 (4)	C14—C13—C12—C11	2.7 (5)
O2—C14—C9—C8	−2.9 (4)	C1—C6—C5—C4	−0.4 (5)
C13—C14—C9—C8	178.7 (3)	C7—C6—C5—C4	179.3 (3)
C10—C9—C8—O1	−31.8 (4)	C6—C5—C4—C3	0.3 (6)
C14—C9—C8—O1	148.1 (3)	C8—N1—C7—C6	−134.0 (3)
C10—C9—C8—N1	144.3 (3)	C5—C6—C7—N1	44.4 (4)
C14—C9—C8—N1	−35.8 (4)	C1—C6—C7—N1	−135.9 (3)
O1—C8—N1—C7	−2.8 (5)	C5—C4—C3—C2	−0.3 (6)
C9—C8—N1—C7	−178.8 (3)	C4—C3—C2—C1	0.4 (7)

C14—O2—C15—C15^{i}	179.5 (3)	C5—C6—C1—C2	0.5 (5)
C14—C9—C10—C11	1.7 (5)	C7—C6—C1—C2	−179.2 (4)
C8—C9—C10—C11	−178.4 (3)	C3—C2—C1—C6	−0.5 (6)
O2—C14—C13—C12	−179.0 (3)		

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···O1^{ii}	0.86	2.19	2.895 (3)	139

Symmetry codes: (ii) *x*+1, *y*, *z*.

Fig. 1

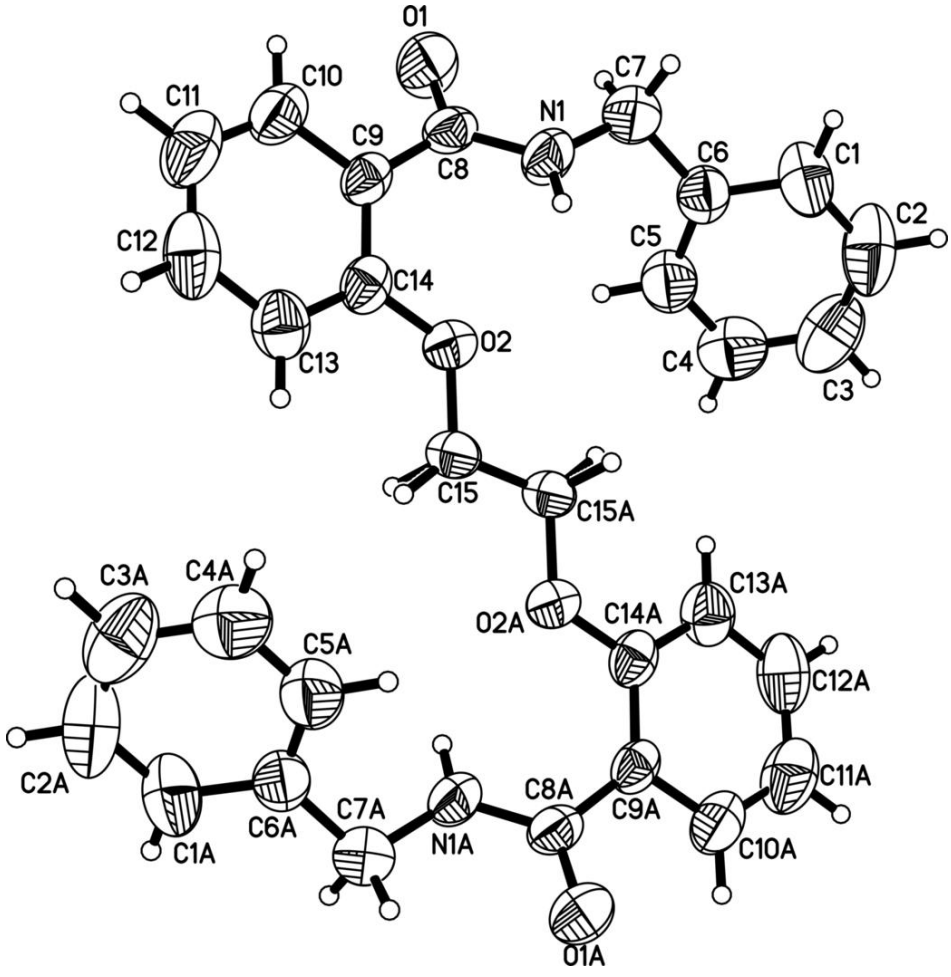
O1
C10
C11
C9
C8
N1
C7
C6
C1
C2
C12
C5
C14
C13
O2
C4
C3
C15
C15A
C3A
C4A
C13A
O2A
C14A
C5A
C12A
C2A
C11A
C8A
C9A
C7A
C6A
N1A
C10A
C1A
O1A

Fig. 2