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N,*N*′-[(1,7-Dioxa-4,10-diazacyclododecane-2,10-diyl)diethylene]-diphthalimide

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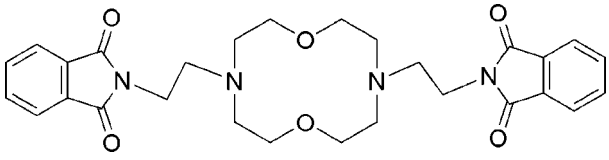
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.051; wR factor = 0.105; data-to-parameter ratio = 14.5.

The title compound, $C_{28}H_{32}N_4O_6$, crystallizes with one half-molecule in the asymmetric unit and the other half generated by an inversion centre. The compound has a crown structure and is an important intermediate for the synthesis of biologically active 1,7-dioxa-4,10-diazacyclododecane derivatives. Intermolecular π–π stacking interactions, with a perpendicular distance of 3.57 Å, involving the planar phthalimide groups and weak C—H···O hydrogen bonds are present.

Related literature

For related literature, see: Aoki & Kimura (2004); Chin & Morrow (1994); Kassai *et al.* (2004); Sheng, Lu, Chen *et al.* (2007); Sheng, Lu, Zhang *et al.* (2007).



Experimental

Crystal data

$C_{28}H_{32}N_4O_6$
M_r = 520.58
Monoclinic, $P2_1/c$
a = 11.014 (13) Å
b = 8.986 (11) Å
c = 13.688 (16) Å
β = 109.746 (18)°
V = 1275 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 (2) K
0.32 × 0.26 × 0.24 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.97, T_{max} = 0.98
6537 measured reflections
2494 independent reflections
1517 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.106
S = 1.03
2494 reflections
172 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···O2^{i}	0.93	2.50	3.244 (5)	137
C9—H9B···O2	0.97	2.47	2.874 (4)	105

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2349).

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Sheng, X., Lu, X.-M., Zhang, J.-J., Chen, Y.-T., Lu, G.-Y., Shao, Y., Liu, F. & Xu, Q. (2007). *J. Org. Chem.* **72**, 1799–1802.

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supplementary materials

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N,*N*'-[(1,7-Dioxa-4,10-diazacyclododecane-2,10-diyl)diethylene]diphthalimide

X. Sheng, D.-H. Wu, Z.-L. Jia, Y. Shao and G.-Y. Lu

Comment

Artificial nucleases have attracted extensive attention due to their potential applications in the fields of molecular biological technology and drug development (Aoki & Kimura, 2004; Chin & Morrow, 1994). The derivatives of aza-crown ethers, such as 1,7-dioxa-4,10-diazacyclododecane and 1,4,7-triazacyclononane exhibit excellent ability to cleave nucleic acids (Sheng, Lu, Chen *et al.*, 2007; Sheng, Lu, Zhang *et al.*, 2007), phosphodiesters, dipeptides and proteins (Kassai *et al.*, 2004). The title compound, (I), is an important intermediate for the synthesis of artificial nucleases 1,7-dioxa-4,10-diazacyclododecane derivatives containing diaminoethyl double side arms, and (I) itself might be a DNA intercalation reagent.

The title molecule (I) is symmetric with an inversion centre imposed at the mid-point of the 1,7-dioxa-4,10-diazacyclododecane ring [symmetry code: (*a*) 1 − *x*, 1 − *y*, −*z*], (Fig. 1). In the title molecule (I), (Fig. 2), the intermolecular π–π stacking interaction of the planar phthalimide groups and weak intermolecular C—H···O hydrogen bonds effectively stabilize the crystal structure.

Experimental

The title compound (I) was synthesized according to the literature procedure (Sheng, Lu, Chen *et al.*, 2007; Sheng, Lu, Zhang *et al.*, 2007). A stirred solution of 1,7-dioxa-4,10-diazacyclododecane (0.26 g, 0.0015 mol), *N*-(2-bromoethyl)phthalimide (0.80 g, 0.0031 mol), and anhydrous potassium carbonate (0.50 g) in dry $CHCl_3$ (25 ml) was heated at 348 K for 12 h. The mixture was then allowed to cool to room temperature and filtered. The filtrate was concentrated under reduced pressure to give a brown oil and purified by silica gel chromatographic column (chloroform/methanol, 2/1 then 1/2) to obtain the title compound (I) as a colourless solid (0.68 mg, 0.0013 mol, yield: 87%, m.p. 481–482 K). Colourless single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution at room temperature. ^{1}H NMR ($CDCl_3$): δ (p.p.m) 2.58 (t, 8H, $4NCH_2$), 2.73 (t, 4H, $2NCH_2$), 3.44 (t, 8H, $4OCH_2$), 3.72 (t, 4H, $2CONCH_2$), 7.68–7.72 (m, 4H, 2ArH), 7.80–7.84 (m, 4H, 2ArH). ^{13}C NMR ($CDCl_3$): δ (p.p.m) 36.18 (NCH_2), 53.82 (NCH_2), 55.33 (NCH_2), 69.65 (OCH_2), 123.50 (Ar C), 132.6 (Ar C), 134.24 (Ar C), 168.79 (C=O). ESI-MS m/*z* $[M+H]^+$ calcd. 525.2, found 525.3.

Refinement

H atoms were located geometrically and allowed to ride on their parent atoms with C—H distances of 0.93–0.97 Å and U_{iso}=1.2U_{eq} of the parent atoms.

Figures

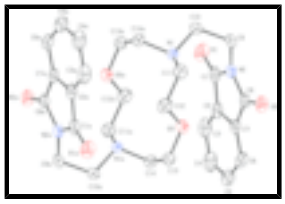

Fig. 1. View of the title compound. Displacement ellipsoids are drawn at the 50% probability level and H atoms are omitted for clarity.

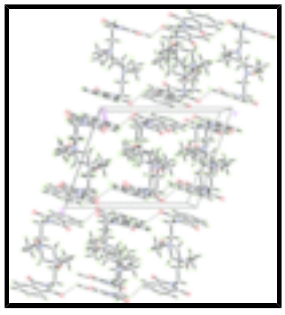

Fig. 2. The molecular packing of (I) viewed along the *b* axis.

_N_,_N_'-[(1,7-Dioxa-4,10-diazacyclododecane-2,10-diyl)diethylene]diphthalimide

Crystal data

$C_{28}H_{32}N_4O_6$	F_{000} = 552
M_r = 520.58	D_x = 1.356 Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: 481-482 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 11.014 (13) Å	Cell parameters from 778 reflections
b = 8.986 (11) Å	θ = 2.9–27.8º
c = 13.688 (16) Å	μ = 0.10 mm^{-1}
β = 109.746 (18)º	T = 293 (2) K
V = 1275 (3) Å^3	Block, colourless
Z = 2	0.32 × 0.26 × 0.24 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer	2494 independent reflections
Radiation source: sealed tube	1517 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.038
T = 293(2) K	θ_{max} = 26.0º
φ and ω scans	θ_{min} = 2.8º
Absorption correction: multi-scan (SADABS; Bruker, 2000)	h = −12→13
T_{min} = 0.97, T_{max} = 0.98	k = −11→7
6537 measured reflections	l = −16→15

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.051$	H-atom parameters constrained
$wR(F^2) = 0.106$	$w = 1/[\sigma^2(F_o^2) + (0.04P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} < 0.001$
2494 reflections	$\Delta\rho_{max} = 0.14$ e Å^{-3}
172 parameters	$\Delta\rho_{min} = -0.13$ 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 $F^2 > 2$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}
C1	0.81133 (17)	0.6171 (2)	−0.02553 (16)	0.0498 (5)
C2	0.82852 (16)	0.4587 (2)	−0.04563 (16)	0.0461 (5)
C3	0.8035 (2)	0.3817 (3)	−0.13666 (17)	0.0634 (7)
H3	0.7731	0.4304	−0.2003	0.076*
C4	0.8245 (2)	0.2308 (3)	−0.1318 (2)	0.0750 (8)
H4	0.8078	0.1767	−0.1929	0.090*
C5	0.8703 (2)	0.1582 (3)	−0.0370 (2)	0.0708 (7)
H5	0.8822	0.0556	−0.0355	0.085*
C6	0.89907 (18)	0.2366 (2)	0.05674 (18)	0.0556 (6)
H6	0.9327	0.1889	0.1205	0.067*
C7	0.87551 (15)	0.3861 (2)	0.04999 (15)	0.0417 (5)
C8	0.88977 (15)	0.4992 (2)	0.13198 (16)	0.0418 (5)
C9	0.82979 (18)	0.7656 (2)	0.13443 (18)	0.0568 (6)
H9A	0.8859	0.8432	0.1248	0.068*
H9B	0.8560	0.7442	0.2081	0.068*
C10	0.69116 (18)	0.8226 (2)	0.09738 (17)	0.0518 (6)
H10A	0.6815	0.8929	0.1481	0.062*
H10B	0.6754	0.8759	0.0326	0.062*

C11	0.5977 (2)	0.6286 (2)	0.17576 (16)	0.0502 (5)
H11A	0.5523	0.6869	0.2121	0.060*
H11B	0.6867	0.6185	0.2210	0.060*
C12	0.5374 (2)	0.4770 (2)	0.15272 (18)	0.0537 (6)
H12A	0.5228	0.4371	0.2137	0.064*
H12B	0.4551	0.4829	0.0968	0.064*
C13	0.5746 (2)	0.2413 (2)	0.08825 (17)	0.0521 (5)
H13A	0.5014	0.2201	0.1102	0.063*
H13B	0.6407	0.1675	0.1194	0.063*
C14	0.53382 (18)	0.2294 (2)	−0.02717 (17)	0.0480 (5)
H14A	0.5970	0.2798	−0.0505	0.058*
H14B	0.5331	0.1253	−0.0460	0.058*
N1	0.59403 (14)	0.70666 (16)	0.08141 (12)	0.0379 (4)
N2	0.84659 (13)	0.63336 (18)	0.08047 (13)	0.0448 (4)
O1	0.77458 (15)	0.71729 (19)	−0.08768 (13)	0.0729 (5)
O2	0.93177 (13)	0.48408 (17)	0.22508 (11)	0.0560 (4)
O3	0.62317 (12)	0.38433 (15)	0.12362 (12)	0.0519 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0270 (9)	0.0660 (15)	0.0500 (12)	−0.0038 (9)	0.0045 (8)	0.0156 (11)
C2	0.0245 (8)	0.0677 (14)	0.0449 (12)	0.0007 (9)	0.0101 (7)	0.0073 (10)
C3	0.0420 (12)	0.103 (2)	0.0427 (14)	0.0035 (12)	0.0111 (10)	−0.0027 (13)
C4	0.0454 (13)	0.111 (2)	0.0666 (18)	0.0093 (14)	0.0164 (12)	−0.0300 (16)
C5	0.0402 (12)	0.0697 (16)	0.097 (2)	0.0149 (11)	0.0160 (12)	−0.0222 (15)
C6	0.0365 (11)	0.0597 (15)	0.0636 (15)	0.0130 (9)	0.0077 (10)	0.0019 (11)
C7	0.0229 (8)	0.0530 (12)	0.0459 (12)	0.0027 (8)	0.0075 (7)	0.0031 (9)
C8	0.0247 (9)	0.0514 (12)	0.0434 (12)	−0.0034 (8)	0.0038 (8)	0.0068 (9)
C9	0.0347 (10)	0.0470 (12)	0.0717 (16)	−0.0077 (9)	−0.0043 (10)	−0.0061 (11)
C10	0.0445 (10)	0.0342 (11)	0.0661 (15)	−0.0044 (9)	0.0047 (10)	−0.0070 (9)
C11	0.0548 (12)	0.0544 (12)	0.0392 (12)	0.0031 (10)	0.0128 (9)	−0.0081 (9)
C12	0.0537 (12)	0.0525 (13)	0.0614 (15)	0.0063 (10)	0.0280 (10)	0.0123 (11)
C13	0.0474 (12)	0.0410 (12)	0.0539 (12)	−0.0009 (9)	−0.0013 (9)	0.0086 (9)
C14	0.0378 (10)	0.0407 (11)	0.0597 (13)	0.0065 (8)	0.0087 (9)	−0.0017 (9)
N1	0.0335 (7)	0.0322 (8)	0.0414 (9)	0.0012 (6)	0.0038 (6)	−0.0020 (7)
N2	0.0294 (7)	0.0478 (10)	0.0484 (10)	−0.0025 (7)	0.0014 (7)	0.0052 (8)
O1	0.0615 (10)	0.0836 (12)	0.0632 (10)	0.0006 (9)	0.0074 (8)	0.0369 (9)
O2	0.0483 (8)	0.0700 (10)	0.0383 (9)	0.0018 (7)	−0.0002 (6)	0.0065 (7)
O3	0.0364 (7)	0.0480 (8)	0.0637 (10)	−0.0032 (6)	0.0073 (6)	−0.0101 (7)

Geometric parameters (Å, °)

C1—O1	1.211 (3)	C9—H9B	0.9700
C1—N2	1.377 (3)	C10—N1	1.456 (3)
C1—C2	1.474 (3)	C10—H10A	0.9700
C2—C3	1.369 (3)	C10—H10B	0.9700
C2—C7	1.396 (3)	C11—N1	1.458 (3)
C3—C4	1.373 (4)	C11—C12	1.502 (3)

C3—H3	0.9300	C11—H11A	0.9700
C4—C5	1.386 (4)	C11—H11B	0.9700
C4—H4	0.9300	C12—O3	1.414 (3)
C5—C6	1.403 (3)	C12—H12A	0.9700
C5—H5	0.9300	C12—H12B	0.9700
C6—C7	1.366 (3)	C13—O3	1.414 (3)
C6—H6	0.9300	C13—C14	1.493 (3)
C7—C8	1.483 (3)	C13—H13A	0.9700
C8—O2	1.207 (3)	C13—H13B	0.9700
C8—N2	1.396 (3)	C14—N1^{i}	1.469 (3)
C9—N2	1.443 (3)	C14—H14A	0.9700
C9—C10	1.526 (3)	C14—H14B	0.9700
C9—H9A	0.9700	N1—C14^{i}	1.469 (3)
O1—C1—N2	124.7 (2)	C9—C10—H10B	108.7
O1—C1—C2	128.4 (2)	H10A—C10—H10B	107.6
N2—C1—C2	106.92 (17)	N1—C11—C12	111.88 (18)
C3—C2—C7	120.9 (2)	N1—C11—H11A	109.2
C3—C2—C1	131.2 (2)	C12—C11—H11A	109.2
C7—C2—C1	107.86 (19)	N1—C11—H11B	109.2
C2—C3—C4	118.5 (2)	C12—C11—H11B	109.2
C2—C3—H3	120.8	H11A—C11—H11B	107.9
C4—C3—H3	120.8	O3—C12—C11	107.63 (18)
C3—C4—C5	120.8 (2)	O3—C12—H12A	110.2
C3—C4—H4	119.6	C11—C12—H12A	110.2
C5—C4—H4	119.6	O3—C12—H12B	110.2
C4—C5—C6	121.2 (3)	C11—C12—H12B	110.2
C4—C5—H5	119.4	H12A—C12—H12B	108.5
C6—C5—H5	119.4	O3—C13—C14	111.77 (17)
C7—C6—C5	116.9 (2)	O3—C13—H13A	109.3
C7—C6—H6	121.5	C14—C13—H13A	109.3
C5—C6—H6	121.5	O3—C13—H13B	109.3
C6—C7—C2	121.7 (2)	C14—C13—H13B	109.3
C6—C7—C8	130.9 (2)	H13A—C13—H13B	107.9
C2—C7—C8	107.45 (19)	N1^{i}—C14—C13	113.66 (17)
O2—C8—N2	125.02 (19)	N1^{i}—C14—H14A	108.8
O2—C8—C7	128.8 (2)	C13—C14—H14A	108.8
N2—C8—C7	106.20 (19)	N1^{i}—C14—H14B	108.8
N2—C9—C10	113.16 (16)	C13—C14—H14B	108.8
N2—C9—H9A	108.9	H14A—C14—H14B	107.7
C10—C9—H9A	108.9	C10—N1—C11	114.34 (16)
N2—C9—H9B	108.9	C10—N1—C14^{i}	109.05 (16)
C10—C9—H9B	108.9	C11—N1—C14^{i}	111.77 (16)
H9A—C9—H9B	107.8	C1—N2—C8	111.53 (17)
N1—C10—C9	114.29 (18)	C1—N2—C9	125.97 (18)
N1—C10—H10A	108.7	C8—N2—C9	122.20 (19)
C9—C10—H10A	108.7	C13—O3—C12	114.87 (17)
N1—C10—H10B	108.7		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···O2^{ii}	0.93	2.50	3.244 (5)	137
C9—H9B···O2	0.97	2.47	2.874 (4)	105

Symmetry codes: (ii) *x*, −*y*+1/2, *z*−1/2.

Fig. 1

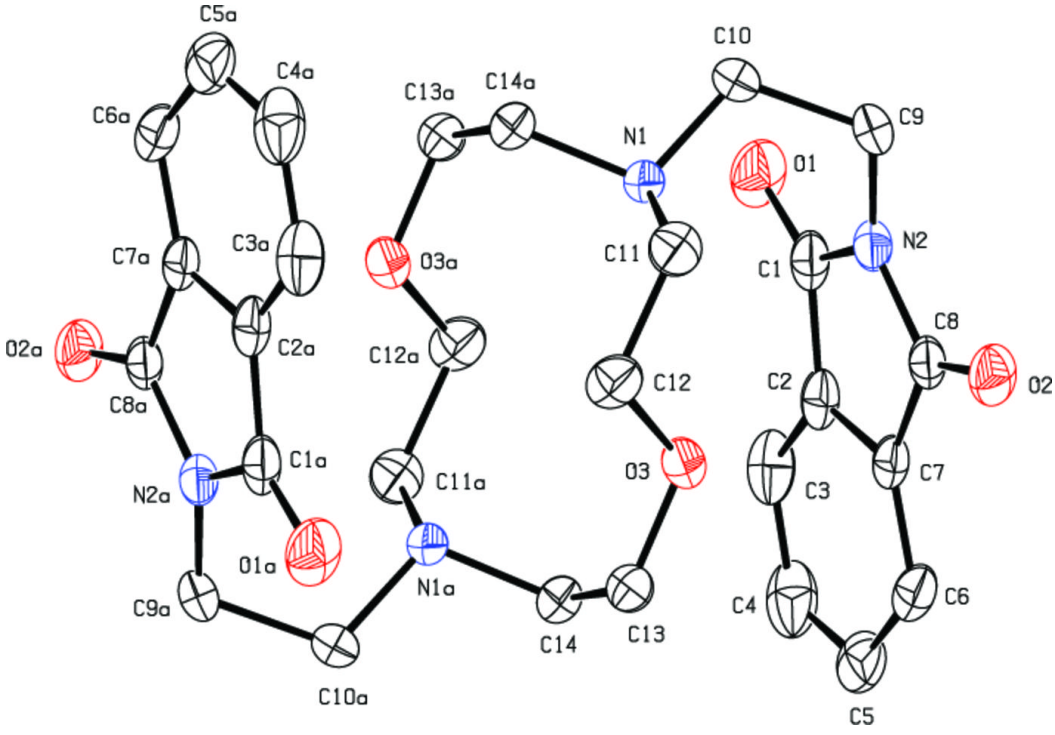
C5a
C4a
C6a
C13a
C14a
N1
C10
C9
O1
C7a
C3a
O3a
C11
C1
N2
C8
O2a
C2a
C12a
C12
C2
O2
C8a
C1a
O3
C11a
C3
C7
N2a
O1a
N1a
C4
C6
C9a
C14
C13
C10a
C5

Fig. 2