30,31-Dimethyl-3,26-dioxa-11,18-diaza-pentacyclo[26.4.0.0^{4,9}.0^{12,17}.0^{20,25}]dotriconta-1(32),4,6,8,12,14,16,20,22,24,28,30-dodecaene-10,19-dione

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Key indicators

Single-crystal X-ray study
$T = 293$ K
Mean σ(C–C) = 0.003 Å
R factor = 0.056
wR factor = 0.166
Data-to-parameter ratio = 17.6

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

The title compound, $C_{30}H_{26}N_2O_4$, crystallizes in space group $P2_1/n$, with two crystallographically independent molecules in the asymmetric unit. Two of the phenyl rings are perpendicular to one another. The molecular structure is influenced by strong N—H···O, N—H···N and C—H···O intramolecular interactions, and the crystal structure is stabilized by C—H···O and C—H···π intermolecular interactions.

Comment

Cyclophane derivatives constitute a novel building block for the potent human immuno deficiency virus (HIV) protease inhibitor (Ettmayer *et al.*, 1996). Cyclophane derivatives act catalytically as cholesterol shuttles to accelerate the exchange of free cholesterol between cells and serum lipo proteins (Christian *et al.*, 1999) and act as potential reversal agents of muscle relaxants by chemical chelation (Cameron *et al.*, 2002). The importance of cyclophane derivatives prompted us to undertake the structure analysis of the title compound, (I).
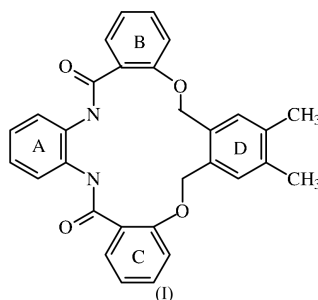

The asymmetric unit of (I) contains two molecules (*A* and *B*), with similar bond lengths and angles.

The C—C bond lengths in the phenyl rings (*A*, *B*, *C* and *D*) are comparable to the reported literature value of 1.384 (13) Å. All of the C—N bond lengths in both of the molecules also agree with the reported literature values [C_{sp2}—N = 1.339 (16) Å and C_{phenyl}—N = 1.419 (17) Å; Allen *et al.*, 1987].

The exocyclic angles around atoms C16 and C24 show considerable asymmetry, with the O17—C16—C15 angle [122.4 (2)° for molecule *A* and 122.9 (2)° for molecule *B*] being wider than the O17—C16—C11 angle [117.2 (2)° for molecule *A* and 117.8 (2)° for molecule *B*], and the C19—C24—C25 angle [124.5 (2)° for molecule *A* and 122.6 (2)° for molecule *B*] being wider than the C23—C24—C25 angle [117.3 (2)° for molecule *A* and 118.9 (2)° for molecule *B*]. This asymmetry may be due to the short contacts between H15*A*···H18*B* (2.19 Å), H15*B*···H18*C* (1.90 Å), H18*A*···H25*B* (2.11 Å) and H25*C*···H18*B* (2.14 Å).
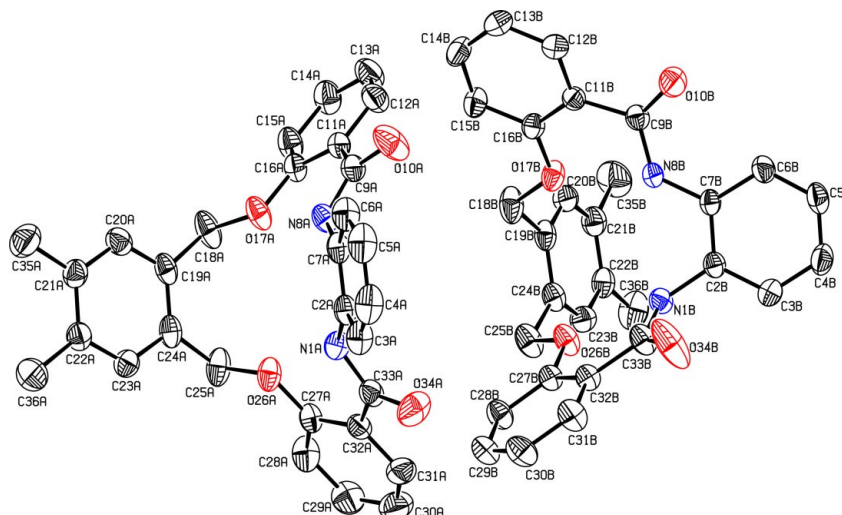
Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids and the atom-labelling scheme. H atoms are omitted for clarity.
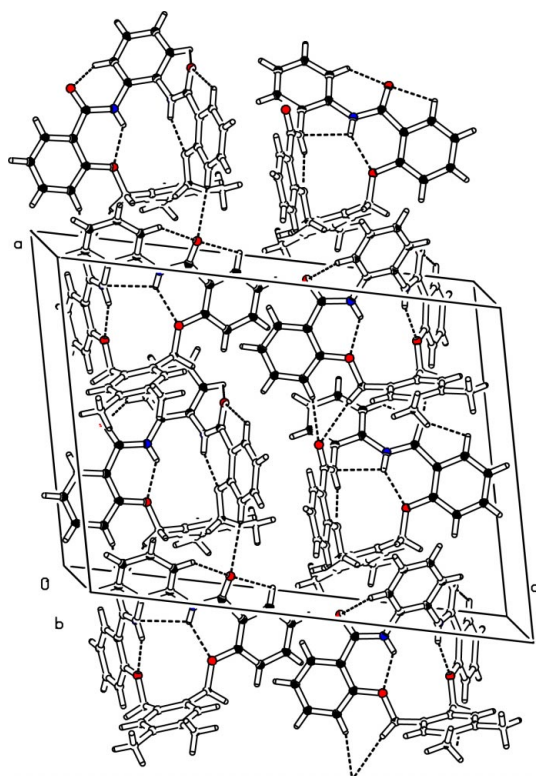
Figure 2
The packing of the molecules of (I), viewed along the *b* axis. Hydrogen bonds are shown as dashed lines.

The values of the C33—N1—C2—C3 [−40.3 (3)° for molecule *A* and 64.6 (3)° for molecule *B*] and C6—C7—N8—C9 [45.2 (3)° for molecule *A* and −13.5 (3)° for molecule *B*] torsion angles indicate that the two amide groups are twisted from the plane of phenyl ring *A*, with dihedral angles of 39.6 (1) and 44.3 (2)° for molecule *A*, and 66.5 (2) and 14.9 (1)° for molecule *B*.

Rings *B* and *D* are perpendicular to one another, the dihedral angle between their planes being 87.0 (1)° for mol-

ecule *A* and 90.0 (1)° for molecule *B*. The dihedral angle between rings *B* and *C* is 84.8 (1)° for molecule *A* and 86.8 (1)° for molecule *B*, indicating that these two phenyl rings are almost perpendicular to one another.

In addition to the van der Waals interactions, the molecular structure is influenced by N—H···O, N—H···N and C—H···O intramolecular interactions. The packing of the molecule is stabilized by C—H···O intermolecular interactions that run along the *ab* plane (Fig. 2). In addition, symmetry-related molecules are also linked by weak C—H···π intermolecular interactions, such that atom H5*B* is 2.91 Å from the centroid of ring *D* at $(\frac{3}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z)$, with a C5—H5*B*···centroid angle of 161° and a C5—H5*B*···centroid distance of 3.801 (3) Å.

Experimental

In high-dilution conditions, benzene 1,2-dioxybis(ethanoyl chloride) (1 mmol) and *ortho*-phenylene diamine (1 mmol) were cyclized in chloroform in the presence of triethylamine, affording the title compound. A single crystal of (I) suitable for X-ray analysis was obtained by recrystallization from chloroform/methanol (1:1) by slow evaporation.

Crystal data

$C_{30}H_{26}N_2O_4$	$D_x = 1.288$ Mg m^{-3}
$M_r = 478.53$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 5463
$a = 15.5767$ (9) Å	reflections
$b = 15.7746$ (9) Å	$\theta = 2.3$–21.7°
$c = 20.5721$ (12) Å	$\mu = 0.09$ mm^{-1}
$\beta = 102.541$ (1)°	$T = 293$ (2) K
$V = 4934.3$ (5) Å^3	Block, colourless
$Z = 8$	$0.24 \times 0.20 \times 0.16$ mm

Data collection

Bruker SMART APEX area-detector diffractometer	$R_{int} = 0.031$
	$\theta_{max} = 28.1°$
ω scans	$h = -19 \rightarrow 20$
30 895 measured reflections	$k = -18 \rightarrow 20$
11 480 independent reflections	$l = -20 \rightarrow 27$
6259 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0872P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.056$	$+ 0.0307P]$
$wR(F^2) = 0.166$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.99$	$(\Delta/\sigma)_{max} < 0.001$
11480 reflections	$\Delta\rho_{max} = 0.30$ e Å^{-3}
653 parameters	$\Delta\rho_{min} = -0.22$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

N1A—C33A	1.342 (3)	N1B—C33B	1.336 (2)
N1A—C2A	1.418 (2)	N1B—C2B	1.434 (2)
C7A—N8A	1.426 (2)	C7B—N8B	1.409 (2)
N8A—C9A	1.348 (3)	N8B—C9B	1.348 (2)
C9A—O10A	1.213 (2)	C9B—O10B	1.218 (2)
C33A—O34A	1.220 (2)	C33B—O34B	1.208 (2)
O17A—C16A—C15A	122.5 (2)	O17B—C16B—C15B	122.9 (2)
O17A—C16A—C11A	117.2 (2)	O17B—C16B—C11B	117.8 (2)
C23A—C24A—C25A	117.3 (2)	C23B—C24B—C25B	118.9 (2)
C19A—C24A—C25A	124.5 (2)	C19B—C24B—C25B	122.6 (2)
C33A—N1A—C2A—C3A	−40.3 (3)	C33B—N1B—C2B—C3B	64.6 (3)
C6A—C7A—N8A—C9A	45.2 (3)	C6B—C7B—N8B—C9B	−13.5 (3)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1A—H1A$\cdots$O26A	0.86	2.01	2.691 (2)	135
N1B—H1B$\cdots$O26B	0.86	2.00	2.662 (2)	134
N8A—H8A$\cdots$O17A	0.86	1.99	2.677 (2)	136
N8B—H8B$\cdots$O17B	0.86	2.10	2.722 (2)	129
N8B—H8B$\cdots$N1B	0.86	2.42	2.791 (3)	106
C3A—H3A$\cdots$O34A	0.93	2.41	2.809 (4)	106
C6A—H6A$\cdots$O10A	0.93	2.53	2.866 (4)	102
C6B—H6B$\cdots$O10B	0.93	2.24	2.820 (3)	120
C12A—H12A$\cdots$O10A	0.93	2.34	2.694 (3)	102
C12B—H12B$\cdots$O10B	0.93	2.38	2.719 (3)	101
C15A—H15A$\cdots$O10B	0.93	2.59	3.517 (3)	173
C28A—H28A$\cdots$O34B	0.93	2.25	3.132 (3)	156
C15B—H15B$\cdots$O10A^i	0.93	2.49	3.376 (3)	158
C18B—H18C$\cdots$O10A^i	0.97	2.33	3.233 (3)	154
C25B—H25C$\cdots$O34A^i	0.97	2.59	3.052 (3)	109

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

H atoms were positioned geometrically and were treated as riding on their parent atoms, with C—H distances of 0.93 (aromatic), 0.96 (methyl) and 0.97 Å (ethylene), N—H distances of 0.86 Å, and U_{iso}(H) values of $1.5U_{eq}$(C) for methyl H atoms and $1.2U_{eq}$(N,C) for other H atoms. The methyl groups were allowed to rotate but not to tip.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1995).

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