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5′-Benzylidene-1″-methyl-4″-phenyltrispiro[1,3-dioxolane-2,1′-cyclohexane-3′,3″-pyrrolidine-2″,3‴-indole]-4′,2‴-dione

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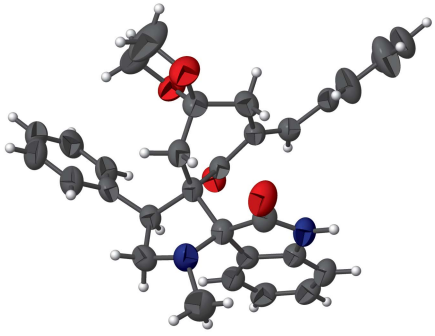
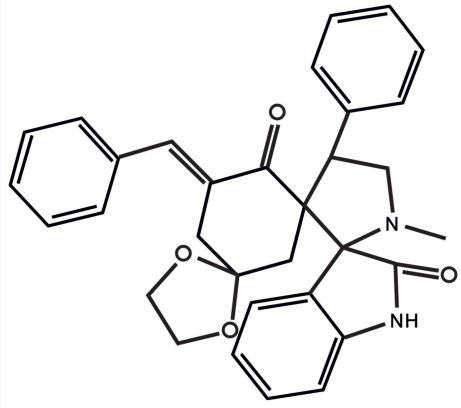
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{32}H_{30}N_2O_4$, two spiro links connect the methyl-substituted pyrrolidine ring to the oxindole and cyclohexanone rings. The cyclohexanone ring is further connected to the dioxalane ring by a third spiro junction. Both the pyrrolidine and dioxalane rings adopt a twist conformation. The indole ring is nearly planar, with a maximum deviation of 0.0296 (7) Å, and the cyclohexanone ring adopts a distorted boat conformation. In the crystal, C—H···O and N—H···N hydrogen-bonding interactions connect molecules into chains running parallel to the *b* axis, which are further linked into layers parallel to the *ab* plane by C—H···O hydrogen bonds.

3D view



Chemical scheme



Structure description

Spiro compounds are present in many natural products (James *et al.*, 1991) and possess anticancer (Chin *et al.*, 2008), antibacterial (Sar *et al.*, 2006), antioxidant (Sarma *et al.*, 2010) and anticonvulsant activities (Obniska & Kamiński, 2006). Some spiro compounds act as pain-relief agents (Frank *et al.*, 2008) and pesticides (Wei *et al.*, 2009). Spiro pyrrolidine derivatives exhibit antimicrobial and neurotoxic properties (Obniska *et al.*, 2006). Oxindole derivatives are found to have antimicrobial (Karki *et al.*, 2011) and antioxidant (Rindhe *et al.*, 2011) properties. Spirooxindole derivatives act as inhibitors of human NK-1 receptor. Alkaloids containing a spiroindolepyrrolidine nucleus are found to be useful in cancer chemotherapy (Fischer *et al.*, 2000). Spiropyrrolidine compounds containing oxindole and cyclohexanone ring systems are found to have antimicrobial and antifungal activities (Raj *et al.*, 2003). Dioxalane moieties play a significant role in

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H34···N1^{i}	0.90 (2)	2.36 (2)	3.251 (2)	174.3 (18)
C4—H4···O1^{ii}	0.93	2.49	3.340 (2)	152
C11—H11B···O4	0.97	2.27	3.045 (2)	137
C22—H22···O4^{i}	0.93	2.43	3.302 (2)	157
C32—H32···O4^{iii}	0.93	2.59	3.504 (3)	167

Symmetry codes: (i) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (ii) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (iii) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$.

stabilizing mutant HIV-1 RT and nucleoside triphosphate and act as nucleoside reverse transcriptase inhibitors (NRTIs) (Liang *et al.*, 2006).

In the title compound (Fig. 1) the methyl-substituted pyrrolidine ring (N1/C10/C16/C17/C8) and the dioxalane ring (O3/O2/C12/C14/C15) both adopt a twist conformation with puckering parameters q_2 = 0.480 (8) Å, φ_2 = 313.34 (6)° and q_2 = 0.221 (9) Å, φ_2 = 350.46 (3)°, respectively. The six-membered cyclohexanone ring (C8–C13) adopts a distorted boat conformation [Q_T = 0.666 (2) Å, θ = 99.42 (16)°, φ = 71.38 (7)°]. The pyrrolidine ring mean plane forms dihedral angles of 58.10 (1) and 87.45 (9)°, respectively, with the mean planes of the benzene ring and the mean plane of the cyclohexanone rings The cyclohexanone ring makes a dihedral angle of 84.14 (1)° with the mean plane of the dioxalane ring. The indole ring (C21–C26/N2/C19/C20) is nearly planar with a maximum deviation of 0.0296 (7) Å for atom C23. An

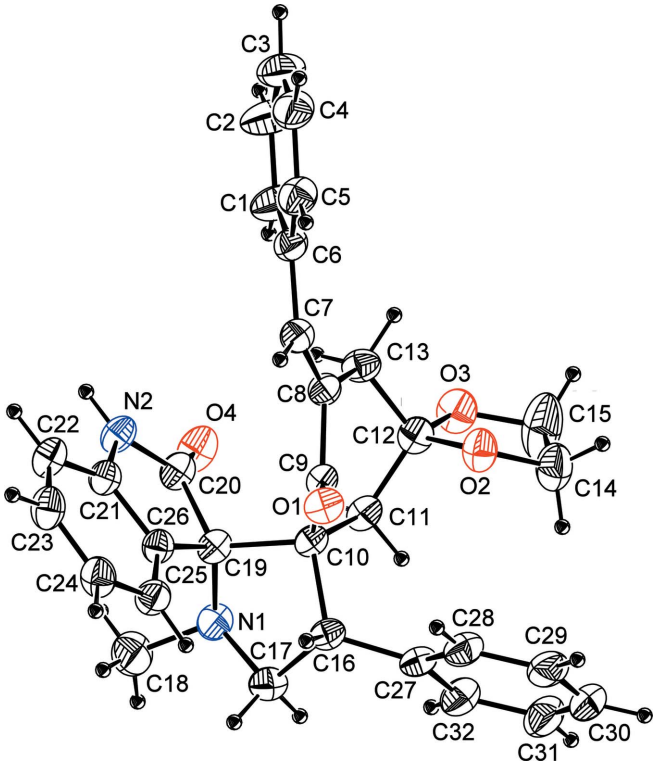


Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 30% probability level.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{32}H_{30}N_2O_4$
M_r	506.58
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	296
a, b, c (Å)	13.9601 (2), 11.1172 (2), 16.7313 (3)
β (°)	96.019 (1)
V (Å^3)	2582.34 (8)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	0.23 × 0.15 × 0.11
Data collection	
Diffractometer	Bruker SMART APEXII area-detector
Absorption correction	Multi-scan (*SADABS*; Bruker, 2008)
T_{min}, T_{max}	0.744, 0.859
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	24317, 6395, 3887
R_{int}	0.029
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.668
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.045, 0.130, 1.04
No. of reflections	6395
No. of parameters	349
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.18, −0.19

Computer programs: *APEX2* and *SAINT* (Bruker, 2008), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *ORTEP-3 for Windows* (Farrugia, 2012), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

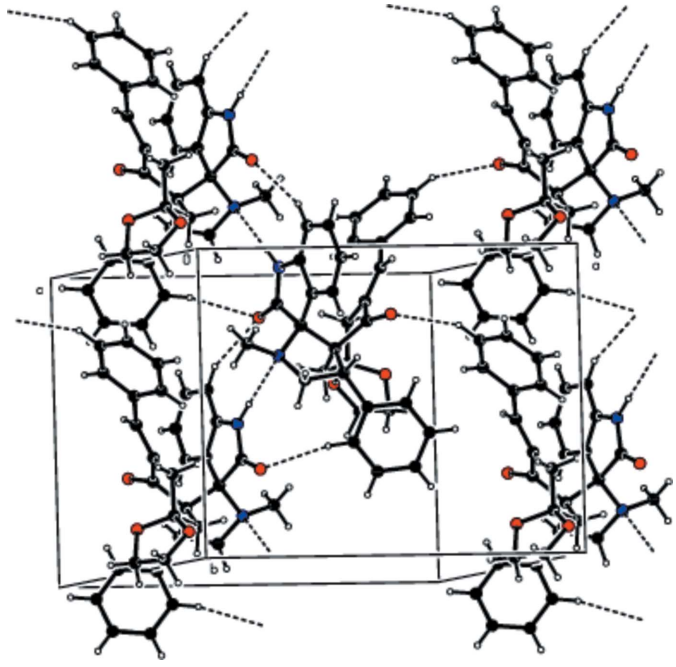


Figure 2
Partial crystal packing of the title compound showing the C—H···O and N—H···N hydrogen bonds (dashed lines), forming chains parallel to the *b* axis. H atoms not involved in hydrogen bonding are omitted for clarity.

intramolecular C—H···O hydrogen bond occurs. The geometrical parameters of the title compound are in good agreement with those previously reported for tris-piropyrrolidine compounds (Chandralekha *et al.*, 2015, 2016).

In the crystal, molecules are linked by weak C—H···O and N—H···O interactions (Table 1), forming chains extending along the *b*-axis direction (Fig. 2). The chains are further linked into layers parallel to the *ab* plane by C—H···O hydrogen bonds.

Synthesis and crystallization

An equimolar mixture of 7,9-bis[(*E*)-benzylidine)]-1,4-dioxo-spiro[4,5]decane-8-one (1 mmol), isatin (1 mmol) and sarcosine (1 mmol) in methanol (25–30 ml) was refluxed for 3 h. After the completion of the reaction as indicated by TLC, the crude product was purified by column chromatography using a mixture of petroleum ether and ethylacetate (8:2 *v*/*v*). Crystals of the title compound were obtained by the slow evaporation of a methanol solution at room temperature.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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full crystallographic data

IUCrData (2017). **2**, x170639 [https://doi.org/10.1107/S2414314617006393]

5′-Benzylidene-1′′-methyl-4′′-phenyltrispiro[1,3-dioxolane-2,1′-cyclohexane-3′,3′′-pyrrolidine-2′′,3′′′-indole]-4′,2′′′-dione

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Crystal data

$C_{32}H_{30}N_2O_4$
M_r = 506.58
Monoclinic, $P2_1/n$
a = 13.9601 (2) Å
b = 11.1172 (2) Å
c = 16.7313 (3) Å
β = 96.019 (1)°
V = 2582.34 (8) Å^3
Z = 4
$F(000)$ = 1072
D_x = 1.303 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6394 reflections
θ = 1.8–28.4°
μ = 0.09 mm^{-1}
T = 296 K
Block, colorless
0.23 × 0.15 × 0.11 mm

Data collection

Bruker SMART APEXII area-detector diffractometer
Radiation source: fine-focus sealed tube
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.744, T_{max} = 0.859
24317 measured reflections
6395 independent reflections
3887 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 28.4°, θ_{min} = 1.8°
h = −18→14
k = −14→14
l = −22→22

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.130
S = 1.04
6395 reflections
349 parameters
0 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0534P)^2 + 0.3939P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Extinction correction: SHELXL2014 (Sheldrick, 2015b), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0017 (6)

Special details

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

Refinement. The N-bound indole H atom was located in a difference Fourier map and refined freely. All other H atoms were placed geometrically and refined using a riding atom approximation, with C–H = 0.93–0.97 \%A, and with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C) for methyl H atoms. A rotating model was used for the methyl group.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
H34	0.2804 (14)	−0.0171 (18)	0.2545 (12)	0.075 (6)*
O1	0.55773 (8)	0.20767 (10)	0.13693 (6)	0.0542 (3)
O2	0.60579 (9)	0.38736 (11)	0.31054 (7)	0.0640 (3)
O3	0.50902 (10)	0.40585 (12)	0.41115 (7)	0.0714 (4)
O4	0.27663 (10)	0.19821 (11)	0.30938 (8)	0.0711 (4)
N1	0.26684 (9)	0.33561 (12)	0.15637 (9)	0.0563 (4)
N2	0.30089 (10)	0.04319 (13)	0.22479 (9)	0.0539 (3)
C1	0.56550 (15)	−0.05286 (18)	0.39691 (13)	0.0785 (6)
H1	0.5130	−0.0036	0.4033	0.094*
C2	0.5949 (2)	−0.1353 (2)	0.45585 (15)	0.1011 (8)
H2	0.5617	−0.1414	0.5010	0.121*
C3	0.67203 (17)	−0.2077 (2)	0.44884 (15)	0.0856 (6)
H3	0.6933	−0.2607	0.4899	0.103*
C4	0.71750 (14)	−0.20157 (17)	0.38123 (15)	0.0763 (6)
H4	0.7690	−0.2526	0.3751	0.092*
C5	0.68774 (13)	−0.12001 (16)	0.32126 (12)	0.0647 (5)
H5	0.7191	−0.1176	0.2749	0.078*
C6	0.61193 (11)	−0.04194 (14)	0.32915 (10)	0.0529 (4)
C7	0.58309 (11)	0.04513 (14)	0.26516 (10)	0.0510 (4)
H7	0.5944	0.0223	0.2135	0.061*
C8	0.54258 (11)	0.15314 (13)	0.27172 (9)	0.0462 (4)
C9	0.51663 (10)	0.22333 (13)	0.19660 (9)	0.0431 (3)
C10	0.43116 (11)	0.31046 (13)	0.19660 (9)	0.0439 (3)
C11	0.43230 (12)	0.37857 (14)	0.27751 (9)	0.0499 (4)
H11A	0.4331	0.4644	0.2671	0.060*
H11B	0.3735	0.3603	0.3012	0.060*
C12	0.51770 (12)	0.34690 (15)	0.33712 (9)	0.0537 (4)
C13	0.52437 (13)	0.21313 (14)	0.34906 (10)	0.0560 (4)
H13A	0.5763	0.1945	0.3903	0.067*
H13B	0.4648	0.1832	0.3666	0.067*
C14	0.62840 (17)	0.4986 (2)	0.34848 (13)	0.0810 (6)
H14A	0.6055	0.5650	0.3140	0.097*
H14B	0.6974	0.5068	0.3619	0.097*
C15	0.5784 (2)	0.4959 (3)	0.42173 (17)	0.1216 (11)
H15A	0.6237	0.4787	0.4683	0.146*
H15B	0.5483	0.5730	0.4298	0.146*

C16	0.42296 (11)	0.39768 (13)	0.12265 (9)	0.0477 (4)
H16	0.4426	0.3513	0.0773	0.057*
C17	0.31406 (12)	0.41924 (16)	0.10477 (11)	0.0585 (4)
H17A	0.2982	0.5018	0.1169	0.070*
H17B	0.2933	0.4036	0.0486	0.070*
C18	0.17083 (14)	0.2983 (2)	0.12196 (15)	0.0858 (6)
H18A	0.1756	0.2573	0.0720	0.129*
H18B	0.1305	0.3678	0.1126	0.129*
H18C	0.1434	0.2451	0.1587	0.129*
C19	0.33456 (11)	0.23636 (14)	0.17605 (9)	0.0478 (4)
C20	0.30084 (11)	0.16007 (15)	0.24632 (10)	0.0527 (4)
C21	0.32460 (11)	0.02762 (14)	0.14589 (9)	0.0478 (4)
C22	0.33136 (13)	−0.07893 (15)	0.10525 (11)	0.0602 (4)
H22	0.3184	−0.1521	0.1288	0.072*
C23	0.35801 (13)	−0.07359 (16)	0.02831 (11)	0.0633 (5)
H23	0.3651	−0.1444	0.0000	0.076*
C24	0.37418 (13)	0.03456 (17)	−0.00690 (10)	0.0619 (5)
H24	0.3911	0.0363	−0.0592	0.074*
C25	0.36575 (13)	0.14113 (16)	0.03418 (10)	0.0578 (4)
H25	0.3757	0.2142	0.0093	0.069*
C26	0.34236 (11)	0.13880 (14)	0.11277 (9)	0.0472 (4)
C27	0.48698 (12)	0.50767 (14)	0.13050 (9)	0.0513 (4)
C28	0.57991 (13)	0.49828 (17)	0.10901 (11)	0.0620 (5)
H28	0.5999	0.4264	0.0877	0.074*
C29	0.64358 (15)	0.5941 (2)	0.11874 (12)	0.0752 (6)
H29	0.7059	0.5856	0.1047	0.090*
C30	0.61510 (18)	0.70108 (19)	0.14885 (13)	0.0801 (6)
H30	0.6579	0.7652	0.1557	0.096*
C31	0.52327 (18)	0.71291 (18)	0.16884 (13)	0.0813 (6)
H31	0.5035	0.7858	0.1888	0.098*
C32	0.45946 (15)	0.61805 (16)	0.15977 (12)	0.0674 (5)
H32	0.3971	0.6280	0.1734	0.081*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0603 (7)	0.0571 (7)	0.0482 (6)	0.0074 (5)	0.0201 (5)	−0.0006 (5)
O2	0.0668 (8)	0.0686 (8)	0.0587 (7)	−0.0144 (6)	0.0169 (6)	−0.0134 (6)
O3	0.0961 (10)	0.0747 (8)	0.0465 (7)	−0.0110 (7)	0.0218 (6)	−0.0175 (6)
O4	0.0849 (9)	0.0665 (8)	0.0694 (8)	−0.0102 (6)	0.0441 (7)	−0.0104 (6)
N1	0.0473 (8)	0.0520 (8)	0.0710 (9)	0.0041 (6)	0.0122 (7)	0.0024 (7)
N2	0.0617 (8)	0.0473 (8)	0.0562 (8)	−0.0055 (6)	0.0232 (7)	0.0015 (6)
C1	0.0779 (13)	0.0704 (12)	0.0926 (15)	0.0236 (10)	0.0340 (11)	0.0289 (11)
C2	0.125 (2)	0.0888 (16)	0.0965 (17)	0.0344 (15)	0.0462 (15)	0.0448 (13)
C3	0.0870 (15)	0.0738 (14)	0.0952 (16)	0.0150 (11)	0.0054 (13)	0.0334 (12)
C4	0.0584 (11)	0.0577 (11)	0.1112 (17)	0.0115 (9)	0.0019 (11)	0.0116 (11)
C5	0.0608 (11)	0.0547 (10)	0.0798 (13)	0.0059 (8)	0.0131 (9)	0.0003 (9)
C6	0.0486 (9)	0.0457 (9)	0.0642 (10)	0.0006 (7)	0.0048 (8)	0.0056 (7)

C7	0.0488 (9)	0.0500 (9)	0.0546 (9)	0.0010 (7)	0.0076 (7)	0.0013 (7)
C8	0.0473 (8)	0.0463 (9)	0.0459 (8)	−0.0016 (7)	0.0097 (7)	0.0028 (7)
C9	0.0476 (8)	0.0397 (8)	0.0435 (8)	−0.0018 (6)	0.0113 (7)	−0.0023 (6)
C10	0.0500 (8)	0.0405 (8)	0.0432 (8)	0.0011 (6)	0.0139 (7)	0.0009 (6)
C11	0.0624 (10)	0.0414 (8)	0.0488 (9)	0.0006 (7)	0.0191 (7)	−0.0027 (7)
C12	0.0666 (10)	0.0566 (10)	0.0404 (8)	−0.0044 (8)	0.0173 (7)	−0.0059 (7)
C13	0.0693 (11)	0.0558 (10)	0.0441 (9)	0.0043 (8)	0.0119 (8)	0.0047 (7)
C14	0.0905 (15)	0.0779 (13)	0.0751 (13)	−0.0227 (11)	0.0114 (11)	−0.0172 (11)
C15	0.156 (3)	0.120 (2)	0.0979 (19)	−0.0632 (19)	0.0531 (18)	−0.0549 (16)
C16	0.0562 (9)	0.0433 (8)	0.0450 (8)	0.0043 (7)	0.0120 (7)	0.0023 (6)
C17	0.0593 (10)	0.0574 (10)	0.0589 (10)	0.0069 (8)	0.0072 (8)	0.0074 (8)
C18	0.0533 (11)	0.0822 (14)	0.1205 (19)	0.0042 (10)	0.0019 (11)	0.0063 (13)
C19	0.0497 (9)	0.0455 (8)	0.0504 (9)	0.0013 (7)	0.0155 (7)	−0.0006 (7)
C20	0.0496 (9)	0.0541 (10)	0.0577 (10)	−0.0018 (7)	0.0213 (8)	−0.0009 (8)
C21	0.0452 (8)	0.0493 (9)	0.0503 (9)	−0.0027 (7)	0.0117 (7)	−0.0012 (7)
C22	0.0674 (11)	0.0469 (9)	0.0683 (11)	−0.0082 (8)	0.0175 (9)	−0.0052 (8)
C23	0.0685 (11)	0.0583 (11)	0.0646 (11)	−0.0092 (9)	0.0143 (9)	−0.0183 (9)
C24	0.0699 (11)	0.0695 (12)	0.0473 (9)	−0.0115 (9)	0.0113 (8)	−0.0110 (8)
C25	0.0724 (11)	0.0552 (10)	0.0464 (9)	−0.0079 (8)	0.0082 (8)	−0.0003 (8)
C26	0.0468 (8)	0.0480 (9)	0.0476 (9)	−0.0030 (7)	0.0086 (7)	−0.0020 (7)
C27	0.0604 (10)	0.0468 (9)	0.0477 (9)	0.0020 (7)	0.0112 (7)	0.0121 (7)
C28	0.0660 (11)	0.0575 (10)	0.0648 (11)	0.0005 (9)	0.0172 (9)	0.0160 (8)
C29	0.0698 (12)	0.0789 (14)	0.0778 (13)	−0.0103 (10)	0.0122 (10)	0.0303 (11)
C30	0.0956 (17)	0.0639 (13)	0.0779 (14)	−0.0256 (12)	−0.0035 (12)	0.0208 (11)
C31	0.1030 (17)	0.0495 (11)	0.0910 (15)	−0.0065 (11)	0.0088 (13)	0.0022 (10)
C32	0.0745 (12)	0.0492 (10)	0.0798 (13)	0.0032 (9)	0.0150 (10)	0.0040 (9)

Geometric parameters (Å, º)

O1—C9	1.2149 (16)	C13—H13B	0.9700
O2—C14	1.411 (2)	C14—C15	1.473 (3)
O2—C12	1.424 (2)	C14—H14A	0.9700
O3—C15	1.391 (3)	C14—H14B	0.9700
O3—C12	1.4176 (18)	C15—H15A	0.9700
O4—C20	1.2174 (19)	C15—H15B	0.9700
N1—C18	1.462 (2)	C16—C27	1.512 (2)
N1—C19	1.468 (2)	C16—C17	1.537 (2)
N1—C17	1.471 (2)	C16—H16	0.9800
N2—C20	1.348 (2)	C17—H17A	0.9700
N2—C21	1.405 (2)	C17—H17B	0.9700
N2—H34	0.90 (2)	C18—H18A	0.9600
C1—C6	1.369 (2)	C18—H18B	0.9600
C1—C2	1.377 (3)	C18—H18C	0.9600
C1—H1	0.9300	C19—C26	1.528 (2)
C2—C3	1.359 (3)	C19—C20	1.562 (2)
C2—H2	0.9300	C21—C22	1.374 (2)
C3—C4	1.356 (3)	C21—C26	1.388 (2)
C3—H3	0.9300	C22—C23	1.378 (2)

C4—C5	1.384 (3)	C22—H22	0.9300
C4—H4	0.9300	C23—C24	1.368 (2)
C5—C6	1.386 (2)	C23—H23	0.9300
C5—H5	0.9300	C24—C25	1.381 (2)
C6—C7	1.468 (2)	C24—H24	0.9300
C7—C8	1.337 (2)	C25—C26	1.388 (2)
C7—H7	0.9300	C25—H25	0.9300
C8—C9	1.491 (2)	C27—C28	1.386 (2)
C8—C13	1.501 (2)	C27—C32	1.390 (2)
C9—C10	1.537 (2)	C28—C29	1.386 (3)
C10—C11	1.550 (2)	C28—H28	0.9300
C10—C16	1.567 (2)	C29—C30	1.367 (3)
C10—C19	1.587 (2)	C29—H29	0.9300
C11—C12	1.514 (2)	C30—C31	1.365 (3)
C11—H11A	0.9700	C30—H30	0.9300
C11—H11B	0.9700	C31—C32	1.379 (3)
C12—C13	1.502 (2)	C31—H31	0.9300
C13—H13A	0.9700	C32—H32	0.9300

C14—O2—C12	107.23 (13)	C14—C15—H15A	110.3
C15—O3—C12	108.77 (14)	O3—C15—H15B	110.3
C18—N1—C19	114.64 (14)	C14—C15—H15B	110.3
C18—N1—C17	113.39 (15)	H15A—C15—H15B	108.6
C19—N1—C17	106.80 (12)	C27—C16—C17	117.02 (13)
C20—N2—C21	112.12 (14)	C27—C16—C10	116.00 (13)
C20—N2—H34	124.1 (12)	C17—C16—C10	103.83 (12)
C21—N2—H34	123.3 (12)	C27—C16—H16	106.4
C6—C1—C2	121.37 (19)	C17—C16—H16	106.4
C6—C1—H1	119.3	C10—C16—H16	106.4
C2—C1—H1	119.3	N1—C17—C16	106.39 (13)
C3—C2—C1	120.8 (2)	N1—C17—H17A	110.5
C3—C2—H2	119.6	C16—C17—H17A	110.5
C1—C2—H2	119.6	N1—C17—H17B	110.5
C4—C3—C2	119.13 (19)	C16—C17—H17B	110.5
C4—C3—H3	120.4	H17A—C17—H17B	108.6
C2—C3—H3	120.4	N1—C18—H18A	109.5
C3—C4—C5	120.44 (18)	N1—C18—H18B	109.5
C3—C4—H4	119.8	H18A—C18—H18B	109.5
C5—C4—H4	119.8	N1—C18—H18C	109.5
C4—C5—C6	121.04 (18)	H18A—C18—H18C	109.5
C4—C5—H5	119.5	H18B—C18—H18C	109.5
C6—C5—H5	119.5	N1—C19—C26	117.99 (13)
C1—C6—C5	117.13 (16)	N1—C19—C20	109.83 (12)
C1—C6—C7	123.03 (15)	C26—C19—C20	100.67 (12)
C5—C6—C7	119.82 (16)	N1—C19—C10	99.85 (11)
C8—C7—C6	128.21 (16)	C26—C19—C10	113.46 (12)
C8—C7—H7	115.9	C20—C19—C10	115.80 (13)
C6—C7—H7	115.9	O4—C20—N2	125.11 (15)

C7—C8—C9	117.92 (14)	O4—C20—C19	126.60 (15)
C7—C8—C13	125.54 (14)	N2—C20—C19	108.28 (13)
C9—C8—C13	116.48 (13)	C22—C21—C26	123.09 (15)
O1—C9—C8	121.57 (13)	C22—C21—N2	127.33 (15)
O1—C9—C10	121.59 (13)	C26—C21—N2	109.57 (13)
C8—C9—C10	116.73 (12)	C21—C22—C23	117.67 (16)
C9—C10—C11	111.79 (12)	C21—C22—H22	121.2
C9—C10—C16	112.48 (12)	C23—C22—H22	121.2
C11—C10—C16	112.37 (12)	C24—C23—C22	120.86 (16)
C9—C10—C19	108.36 (11)	C24—C23—H23	119.6
C11—C10—C19	111.94 (12)	C22—C23—H23	119.6
C16—C10—C19	99.22 (12)	C23—C24—C25	120.88 (16)
C12—C11—C10	113.41 (12)	C23—C24—H24	119.6
C12—C11—H11A	108.9	C25—C24—H24	119.6
C10—C11—H11A	108.9	C24—C25—C26	119.76 (16)
C12—C11—H11B	108.9	C24—C25—H25	120.1
C10—C11—H11B	108.9	C26—C25—H25	120.1
H11A—C11—H11B	107.7	C21—C26—C25	117.68 (14)
O3—C12—O2	106.42 (13)	C21—C26—C19	109.09 (13)
O3—C12—C13	110.57 (13)	C25—C26—C19	133.19 (14)
O2—C12—C13	108.10 (14)	C28—C27—C32	117.30 (16)
O3—C12—C11	109.55 (13)	C28—C27—C16	118.51 (15)
O2—C12—C11	111.54 (13)	C32—C27—C16	124.17 (15)
C13—C12—C11	110.58 (14)	C27—C28—C29	121.18 (18)
C8—C13—C12	109.82 (13)	C27—C28—H28	119.4
C8—C13—H13A	109.7	C29—C28—H28	119.4
C12—C13—H13A	109.7	C30—C29—C28	120.28 (19)
C8—C13—H13B	109.7	C30—C29—H29	119.9
C12—C13—H13B	109.7	C28—C29—H29	119.9
H13A—C13—H13B	108.2	C31—C30—C29	119.42 (19)
O2—C14—C15	104.66 (17)	C31—C30—H30	120.3
O2—C14—H14A	110.8	C29—C30—H30	120.3
C15—C14—H14A	110.8	C30—C31—C32	120.8 (2)
O2—C14—H14B	110.8	C30—C31—H31	119.6
C15—C14—H14B	110.8	C32—C31—H31	119.6
H14A—C14—H14B	108.9	C31—C32—C27	120.97 (19)
O3—C15—C14	107.05 (17)	C31—C32—H32	119.5
O3—C15—H15A	110.3	C27—C32—H32	119.5

C6—C1—C2—C3	0.7 (4)	C17—N1—C19—C26	−78.91 (16)
C1—C2—C3—C4	−2.8 (4)	C18—N1—C19—C20	−66.90 (19)
C2—C3—C4—C5	2.0 (4)	C17—N1—C19—C20	166.60 (13)
C3—C4—C5—C6	0.8 (3)	C18—N1—C19—C10	170.95 (15)
C2—C1—C6—C5	2.1 (3)	C17—N1—C19—C10	44.45 (15)
C2—C1—C6—C7	−179.7 (2)	C9—C10—C19—N1	−164.64 (11)
C4—C5—C6—C1	−2.8 (3)	C11—C10—C19—N1	71.64 (14)
C4—C5—C6—C7	178.88 (16)	C16—C10—C19—N1	−47.12 (13)
C1—C6—C7—C8	30.7 (3)	C9—C10—C19—C26	−38.16 (16)

C5—C6—C7—C8	−151.10 (17)	C11—C10—C19—C26	−161.88 (12)
C6—C7—C8—C9	−177.82 (15)	C16—C10—C19—C26	79.36 (14)
C6—C7—C8—C13	4.9 (3)	C9—C10—C19—C20	77.57 (15)
C7—C8—C9—O1	−25.1 (2)	C11—C10—C19—C20	−46.16 (17)
C13—C8—C9—O1	152.40 (15)	C16—C10—C19—C20	−164.92 (12)
C7—C8—C9—C10	151.08 (14)	C21—N2—C20—O4	174.93 (16)
C13—C8—C9—C10	−31.41 (19)	C21—N2—C20—C19	−3.78 (18)
O1—C9—C10—C11	−142.89 (14)	N1—C19—C20—O4	−48.5 (2)
C8—C9—C10—C11	40.92 (17)	C26—C19—C20—O4	−173.63 (17)
O1—C9—C10—C16	−15.4 (2)	C10—C19—C20—O4	63.6 (2)
C8—C9—C10—C16	168.44 (12)	N1—C19—C20—N2	130.20 (14)
O1—C9—C10—C19	93.29 (16)	C26—C19—C20—N2	5.05 (16)
C8—C9—C10—C19	−82.90 (15)	C10—C19—C20—N2	−117.70 (15)
C9—C10—C11—C12	1.45 (17)	C20—N2—C21—C22	179.89 (16)
C16—C10—C11—C12	−126.14 (14)	C20—N2—C21—C26	0.63 (19)
C19—C10—C11—C12	123.22 (14)	C26—C21—C22—C23	0.6 (3)
C15—O3—C12—O2	11.2 (2)	N2—C21—C22—C23	−178.53 (16)
C15—O3—C12—C13	128.3 (2)	C21—C22—C23—C24	−2.0 (3)
C15—O3—C12—C11	−109.5 (2)	C22—C23—C24—C25	1.1 (3)
C14—O2—C12—O3	−22.49 (19)	C23—C24—C25—C26	1.2 (3)
C14—O2—C12—C13	−141.28 (16)	C22—C21—C26—C25	1.6 (2)
C14—O2—C12—C11	96.94 (16)	N2—C21—C26—C25	−179.14 (14)
C10—C11—C12—O3	−175.50 (12)	C22—C21—C26—C19	−176.40 (15)
C10—C11—C12—O2	66.95 (17)	N2—C21—C26—C19	2.89 (17)
C10—C11—C12—C13	−53.39 (17)	C24—C25—C26—C21	−2.5 (2)
C7—C8—C13—C12	156.55 (16)	C24—C25—C26—C19	174.90 (16)
C9—C8—C13—C12	−20.8 (2)	N1—C19—C26—C21	−124.11 (14)
O3—C12—C13—C8	−174.84 (13)	C20—C19—C26—C21	−4.70 (16)
O2—C12—C13—C8	−58.72 (17)	C10—C19—C26—C21	119.67 (14)
C11—C12—C13—C8	63.64 (17)	N1—C19—C26—C25	58.4 (2)
C12—O2—C14—C15	24.4 (2)	C20—C19—C26—C25	177.77 (17)
C12—O3—C15—C14	3.9 (3)	C10—C19—C26—C25	−57.9 (2)
O2—C14—C15—O3	−17.5 (3)	C17—C16—C27—C28	−150.01 (15)
C9—C10—C16—C27	−82.41 (16)	C10—C16—C27—C28	86.82 (18)
C11—C10—C16—C27	44.81 (17)	C17—C16—C27—C32	31.5 (2)
C19—C10—C16—C27	163.24 (12)	C10—C16—C27—C32	−91.68 (19)
C9—C10—C16—C17	147.76 (13)	C32—C27—C28—C29	2.0 (3)
C11—C10—C16—C17	−85.03 (15)	C16—C27—C28—C29	−176.65 (16)
C19—C10—C16—C17	33.40 (14)	C27—C28—C29—C30	−1.0 (3)
C18—N1—C17—C16	−150.61 (15)	C28—C29—C30—C31	−0.4 (3)
C19—N1—C17—C16	−23.37 (17)	C29—C30—C31—C32	0.7 (3)
C27—C16—C17—N1	−137.40 (14)	C30—C31—C32—C27	0.4 (3)
C10—C16—C17—N1	−8.18 (17)	C28—C27—C32—C31	−1.7 (3)
C18—N1—C19—C26	47.6 (2)	C16—C27—C32—C31	176.86 (17)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H34···N1^{i}	0.90 (2)	2.36 (2)	3.251 (2)	174.3 (18)
C4—H4···O1^{ii}	0.93	2.49	3.340 (2)	152
C11—H11*B*···O4	0.97	2.27	3.045 (2)	137
C22—H22···O4^{i}	0.93	2.43	3.302 (2)	157
C32—H32···O4^{iii}	0.93	2.59	3.504 (3)	167

Symmetry codes: (i) −*x*+1/2, *y*−1/2, −*z*+1/2; (ii) −*x*+3/2, *y*−1/2, −*z*+1/2; (iii) −*x*+1/2, *y*+1/2, −*z*+1/2.