Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

[(2-{[3′,6′-Bis(ethylamino)-2′,7′-dimethyl-3-oxospiro[1*H*-isoindole-1,9′-9*H*-xanthen]-2-yl}ethyl)aminomethyl]phenol. Corrigendum

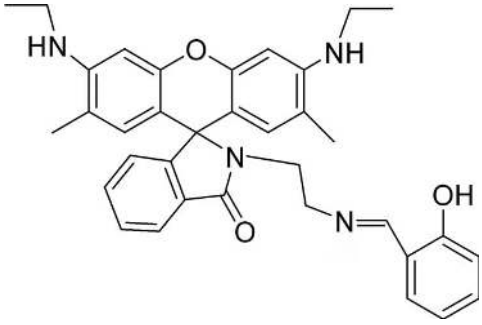
Li-Zhu Zhang, Xiao-Jun Peng,* Shang Gao and Jiang-Li Fan

State Key Laboratory of Fine Chemicals, Dalian University of Technology, 158 Zhongshan Road, Dalian 116012, People's Republic of China
Correspondence e-mail: pengxj@dlut.edu.cn

Received 22 April 2008; accepted 24 April 2008

The title and the chemical diagram of the paper by Zhang, Peng, Gao & Fan [*Acta Cryst.* (2008), E**64**, o403] are corrected.

In the paper by Zhang, Peng, Gao & Fan [*Acta Cryst.* (2008), E**64**, o403], the title and the chemical diagram are incorrect. The correct structure is shown below and the correct title of the original paper should be '[(2-{[3′,6′-Bis(ethylamino)-2′,7′-dimethyl-3-oxospiro[1*H*-isoindole-1,9′-9*H*-xanthen]-2-yl}-ethyl)iminomethyl]phenol'.



Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

[(2-{[3′,6′-Bis(ethylamino)-2′,7′-dimethyl-3-oxospiro[1*H*-isoindole-1,9′-9*H*-xanthen]-2-yl}ethyl)aminomethyl]phenol

Li-Zhu Zhang, Xiao-Jun Peng,* Shang Gao and Jiang-Li Fan

State Key Laboratory of Fine Chemicals, Dalian University of Technology, 158 Zhongshan Road, Dalian 116012, People's Republic of China
Correspondence e-mail: pengxj@dlut.edu.cn

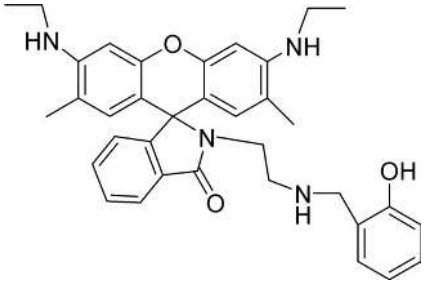
Received 19 November 2007; accepted 29 December 2007

Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.004 Å; R factor = 0.056; wR factor = 0.169; data-to-parameter ratio = 13.1.

The title compound, $C_{35}H_{38}N_4O_3$, was prepared as a spirolactam ring formation of rhodamine dye for comparison with a ring-opened form. The xanthene ring system is approximately planar. The dihedral angles formed by the spirolactam and phenol rings with the xanthene ring system are 85.7 and 109.4°, respectively. Each of the molecules in the crystal structure contains one intramolecular O—H···N hydrogen bond, and they form intermolecular N—H···O hydrogen-bonded chains along the [100] direction. Weak intermolecular C—H···O hydrogen-bonding contacts connect the infinite chains *via* crystallographic inversion centres to form a two-dimensional network.

Related literature

For related literature, see: Kwon *et al.* (2005); Wu *et al.* (2007); De Silva *et al.* (1997).



Experimental

Crystal data

$C_{35}H_{36}N_4O_3$ M_r = 560.68
Triclinic, $P\overline{1}$
a = 11.6453 (5) Å
b = 11.8588 (3) Å
c = 12.9822 (3) Å
α = 116.3210 (10)°
β = 103.173 (2)°
γ = 98.337 (2)°
V = 1501.21 (8) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 273 (2) K
0.30 × 0.30 × 0.25 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.976, T_{max} = 0.980
9518 measured reflections
5018 independent reflections
3667 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.169
S = 1.01
5018 reflections
383 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.55 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3A···N2	0.82	2.01	2.685 (3)	139
N3—H3B···O3^{i}	0.86	2.32	3.144 (1)	160
C9—H9A···O2^{ii}	0.93	2.59	3.468 (3)	157

Symmetry codes: (i) $x+1, y, z$; (ii) $-x+1, -y+3, -z+1$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

This work was supported by the National Natural Science Foundation of China (grant Nos. 20376010 and 20472012) and the Natural Science Foundation of Liaoning Provence (grant No. 20062186).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2058).

References

Bruker (1997). *SADABS* (Version 2.03) and *SHELXTL* (Version 5.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2001). *SAINT-Plus*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2005). *APEX2*. Bruker AXS Inc., Madison, Wisconsin, USA.
De Silva, A. P., Gunaratne, H. Q. N., Gunnlaugsson, T., Huxley, A. J. M., McCoy, C. P., Rademacher, J. T. & Rice, T. E. (1997). *Chem. Rev.* **97**, 1515–1566.
Kwon, J. Y., Jang, Y. J., Lee, Y. J., Kim, K. M., Seo, M. S., Nam, W. & Yoon, J. (2005). *J. Am. Chem. Soc.* **127**, 10107–10111.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Wu, J. S., Hwang, I. C., Kwang, S. K. & Jong, S. K. (2007). *Org. Lett.* **9**, 907–910.

 doi:10.1107/S1600536807068742

supporting information

Acta Cryst. (2008). E**64**, o403 [doi:10.1107/S1600536807068742]

[(2-{[3′,6′-Bis(ethylamino)-2′,7′-dimethyl-3-oxospiro[1*H*-isoindole-1,9′-9*H*-xanthen]-2-yl}ethyl)aminomethyl]phenol

Li-Zhu Zhang, Xiao-Jun Peng, Shang Gao and Jiang-Li Fan

S1. Comment

Rhodamine dyes are widely used in fluorescent labels and other present biological techniques (De Silva *et al.*, 1997). There are a few single-crystal reports about the rhodamine derivatives bearing the lactam moiety (Kwon *et al.*, 2005; Wu *et al.*,2007). Detailed information on their molecular and crystal structures is necessary to understand their photophysical and photochemical properties. As part of our own work on rhodamine dyes, the title compound (Fig. 1) was prepared by the reaction of *N*-(rhodamine-6 G) lactam-ethylenediamine with 2-hydroxybenzaldehyde.

In agreement with other reported models, the main skeleton of the molecule is formed from the xanthene ring and the spirolactam-ring (Kwon *et al.*, 2005; Wu *et al.*, 2007). The atoms of the xanthene ring or the spirolactam-ring are almost coplanar. The planes formed have r.m.s. deviations from the mean plane of only 0.0248 Å for the xanthene ring and 0.0231 Å for the spirolactam-ring, respectively. The dihedral angle between the planes of the xanthene ring and the spirolactam-ring fragment is 85.7°. The phenol part of the molecule (C30 benzene ring plus O3) is close to being planar, with an r.m.s. deviation for the fitted atoms of 0.0065 Å. The dihedral angle with the planes of the xanthene ring and the spirolactam-ring fragment is 109.4° and 107.8° respectively. Each of the molecules in the crystal structure contains one O—H···N intramolecular hydrogen bond, and they form infinite intermolecular N—H···O hydrogen bonded chains along [1 0 0]. Weak intermolecular C—H···O hydrogen bonding contacts connect the infinite chains *via* crystallographic inversion centres to form a two-dimensional network (Table 1).

S2. Experimental

N-(rhodamine-6 G) lactam-ethylenediamine (500 mg, 1.2 mmol) was dissolved in 20 ml of methanol, followed by addition of 2-hydroxybenzaldehyde (146 mg, 1.2 mmol). The yellow solution was mixed for 1 h at room temperature, and then added $NaBH_4$ until the reactant disappeared on TLC. The solvent was removed by rotatory evaporation. The resulting crude product was purified by column chromatography to give 317 mg of the title compound (white solid) in 47.1% yield. Single crystals suitable for X-ray measurements were obtained from hexane/dichloromethane (1:1, *v/v*) solution by slow evaporation at room temperature.

S3. Refinement

The H atoms attached to C, N and O atoms were placed in geometrically calculated positions (C—H = 0.93–0.97 Å, N—H = 0.86 Å and O—H = 0.82 Å) and refined as riding, with U_{iso}(H) = 1.2U_{eq} (C, N) or 1.5U_{eq} (methyl C, O).

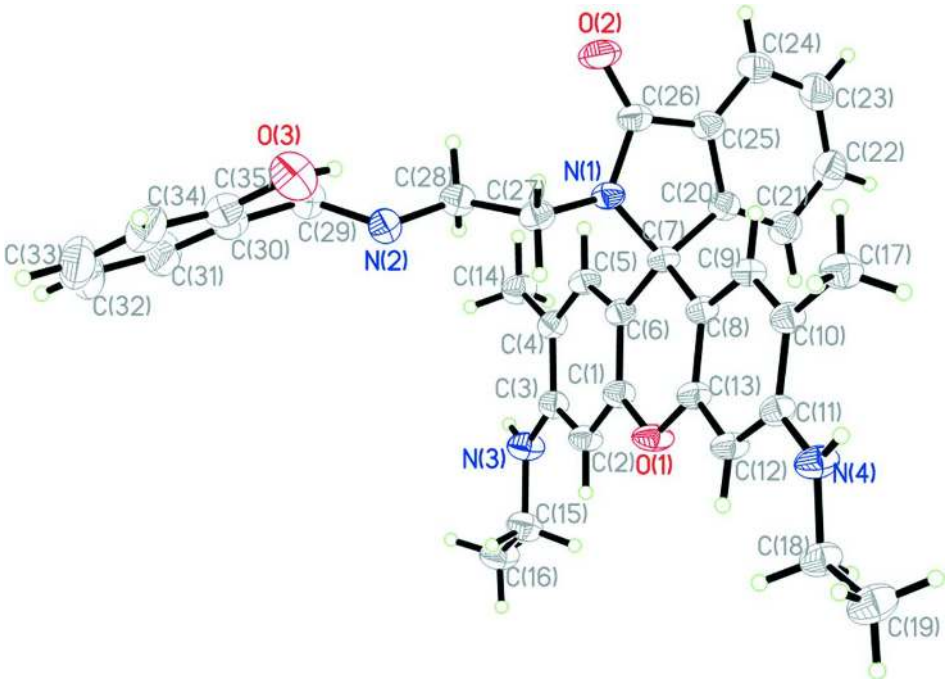


Figure 1

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

[(2-{[3′,6′-Bis(ethylamino)-2′,7′-dimethyl-3-oxospiro[1*H*-isoindole-1,9′-\ 9*H*-xanthen]-2-yl}ethyl)aminomethyl]phenol

Crystal data

$C_{35}H_{36}N_4O_3$
$M_r = 560.68$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 11.6453\ (5)$ Å
$b = 11.8588\ (3)$ Å
$c = 12.9822\ (3)$ Å
$\alpha = 116.321\ (1)°$
$\beta = 103.173\ (2)°$
$\gamma = 98.337\ (2)°$
$V = 1501.21\ (8)$ Å^3
$Z = 2$
$F(000) = 596$
$D_x = 1.240$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2891 reflections
$\theta = 2.7–27.2°$
$\mu = 0.08$ mm^{-1}
$T = 273$ K
Block, white
0.30 × 0.30 × 0.25 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
$T_{min} = 0.976$, $T_{max} = 0.980$
9518 measured reflections
5018 independent reflections
3667 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.7°$
$h = -13\rightarrow12$
$k = -14\rightarrow13$
$l = -15\rightarrow15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.169$
$S = 1.01$
5018 reflections
383 parameters
0 restraints
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0943P)^2 + 0.4133P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.55$ e Å^{-3}
$\Delta\rho_{min} = -0.34$ e Å^{-3}

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.61529 (16)	0.99784 (14)	0.12430 (14)	0.0552 (4)
O2	0.52746 (17)	1.55773 (17)	0.38418 (16)	0.0697 (5)
O3	0.10319 (17)	1.2888 (2)	−0.06993 (17)	0.0752 (6)
H3A	0.1732	1.3180	−0.0213	0.113*
N1	0.55473 (16)	1.35526 (16)	0.27576 (15)	0.0430 (4)
N2	0.34153 (17)	1.29474 (18)	−0.01746 (17)	0.0496 (5)
N3	0.88189 (19)	1.10924 (19)	−0.06256 (18)	0.0571 (5)
H3B	0.9285	1.1680	−0.0682	0.068*
N4	0.35515 (18)	0.83845 (18)	0.29282 (19)	0.0539 (5)
H4A	0.3082	0.8541	0.3369	0.065*
C1	0.6847 (2)	1.0985 (2)	0.11824 (19)	0.0440 (5)
C2	0.7442 (2)	1.0584 (2)	0.0336 (2)	0.0477 (6)
H2B	0.7345	0.9694	−0.0143	0.057*
C3	0.8187 (2)	1.1487 (2)	0.0184 (2)	0.0443 (5)
C4	0.82894 (19)	1.2838 (2)	0.0902 (2)	0.0439 (5)
C5	0.7681 (2)	1.3186 (2)	0.1733 (2)	0.0442 (5)
H5A	0.7758	1.4072	0.2205	0.053*
C6	0.69504 (19)	1.2290 (2)	0.19164 (19)	0.0413 (5)
C7	0.63690 (19)	1.2743 (2)	0.29058 (19)	0.0407 (5)
C8	0.56535 (19)	1.1577 (2)	0.29171 (19)	0.0413 (5)
C9	0.5015 (2)	1.1743 (2)	0.37370 (19)	0.0448 (5)
H9A	0.5057	1.2593	0.4294	0.054*
C10	0.4326 (2)	1.0719 (2)	0.3771 (2)	0.0448 (5)
C11	0.4261 (2)	0.9423 (2)	0.2921 (2)	0.0441 (5)
C12	0.4884 (2)	0.9238 (2)	0.2102 (2)	0.0477 (6)
H12A	0.4847	0.8392	0.1540	0.057*
C13	0.5566 (2)	1.0302 (2)	0.21049 (19)	0.0432 (5)
C14	0.9068 (2)	1.3854 (2)	0.0759 (2)	0.0593 (7)
H14A	0.8967	1.4698	0.1244	0.089*
H14B	0.8818	1.3633	−0.0080	0.089*
H14C	0.9916	1.3879	0.1023	0.089*

C15	0.8734 (3)	0.9726 (2)	−0.1390 (2)	0.0617 (7)
H15A	0.7899	0.9267	−0.1946	0.074*
H15B	0.8922	0.9328	−0.0884	0.074*
C16	0.9601 (3)	0.9574 (3)	−0.2109 (3)	0.0731 (8)
H16A	0.9549	0.8663	−0.2572	0.110*
H16B	1.0426	1.0050	−0.1563	0.110*
H16C	0.9382	0.9914	−0.2651	0.110*
C17	0.3645 (2)	1.0961 (2)	0.4661 (2)	0.0599 (7)
H17A	0.3775	1.1887	0.5149	0.090*
H17B	0.3941	1.0612	0.5177	0.090*
H17C	0.2783	1.0540	0.4229	0.090*
C18	0.3572 (2)	0.7054 (2)	0.2223 (2)	0.0558 (6)
H18A	0.4406	0.6995	0.2449	0.067*
H18B	0.3307	0.6794	0.1366	0.067*
C19	0.2740 (3)	0.6137 (3)	0.2434 (3)	0.0710 (8)
H19A	0.2798	0.5262	0.1987	0.106*
H19B	0.1907	0.6153	0.2162	0.106*
H19C	0.2986	0.6411	0.3286	0.106*
C20	0.7308 (2)	1.3743 (2)	0.41426 (19)	0.0440 (5)
C21	0.8369 (2)	1.3621 (2)	0.4758 (2)	0.0566 (6)
H21A	0.8599	1.2852	0.4427	0.068*
C22	0.9085 (3)	1.4675 (3)	0.5882 (3)	0.0679 (8)
H22A	0.9807	1.4613	0.6312	0.081*
C23	0.8745 (3)	1.5813 (3)	0.6375 (2)	0.0728 (8)
H23A	0.9235	1.6504	0.7138	0.087*
C24	0.7691 (3)	1.5944 (2)	0.5756 (2)	0.0646 (7)
H24A	0.7464	1.6715	0.6087	0.077*
C25	0.6977 (2)	1.4891 (2)	0.4624 (2)	0.0492 (6)
C26	0.5851 (2)	1.4766 (2)	0.3749 (2)	0.0497 (6)
C27	0.4445 (2)	1.3027 (2)	0.1728 (2)	0.0482 (6)
H27A	0.3760	1.3217	0.2018	0.058*
H27B	0.4277	1.2082	0.1272	0.058*
C28	0.4528 (2)	1.3576 (2)	0.0890 (2)	0.0518 (6)
H28A	0.4637	1.4514	0.1323	0.062*
H28B	0.5237	1.3433	0.0629	0.062*
C29	0.3371 (2)	1.3596 (2)	−0.0916 (2)	0.0552 (6)
H29A	0.3996	1.4300	−0.0749	0.066*
C30	0.2200 (2)	1.2987 (2)	−0.1972 (2)	0.0526 (6)
C31	0.2189 (3)	1.2780 (3)	−0.3105 (3)	0.0811 (9)
H31A	0.2927	1.2992	−0.3230	0.097*
C32	0.1091 (5)	1.2257 (4)	−0.4065 (3)	0.1077 (13)
H32A	0.1095	1.2120	−0.4827	0.129*
C33	0.0009 (4)	1.1948 (4)	−0.3886 (4)	0.1007 (12)
H33A	−0.0724	1.1579	−0.4533	0.121*
C34	−0.0008 (3)	1.2176 (3)	−0.2758 (3)	0.0790 (9)
H34A	−0.0752	1.1992	−0.2633	0.095*
C35	0.1079 (2)	1.2677 (2)	−0.1814 (2)	0.0554 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0714 (11)	0.0304 (8)	0.0645 (10)	0.0073 (7)	0.0418 (9)	0.0170 (7)
O2	0.0791 (13)	0.0448 (11)	0.0760 (12)	0.0301 (9)	0.0223 (10)	0.0201 (9)
O3	0.0627 (12)	0.0906 (14)	0.0746 (12)	0.0092 (10)	0.0367 (10)	0.0397 (11)
N1	0.0433 (10)	0.0308 (9)	0.0472 (10)	0.0084 (7)	0.0125 (8)	0.0147 (8)
N2	0.0487 (11)	0.0499 (11)	0.0488 (10)	0.0077 (8)	0.0154 (9)	0.0255 (9)
N3	0.0664 (14)	0.0418 (11)	0.0643 (12)	0.0082 (9)	0.0374 (11)	0.0220 (9)
N4	0.0567 (12)	0.0400 (11)	0.0742 (13)	0.0135 (9)	0.0381 (11)	0.0281 (10)
C1	0.0464 (13)	0.0336 (12)	0.0499 (12)	0.0046 (9)	0.0183 (10)	0.0197 (10)
C2	0.0537 (14)	0.0333 (12)	0.0498 (12)	0.0064 (10)	0.0215 (11)	0.0150 (10)
C3	0.0421 (12)	0.0404 (12)	0.0481 (12)	0.0070 (9)	0.0154 (10)	0.0212 (10)
C4	0.0391 (12)	0.0396 (12)	0.0514 (12)	0.0053 (9)	0.0126 (10)	0.0240 (10)
C5	0.0444 (12)	0.0312 (11)	0.0534 (13)	0.0080 (9)	0.0160 (10)	0.0189 (10)
C6	0.0406 (12)	0.0339 (12)	0.0462 (12)	0.0068 (9)	0.0131 (10)	0.0188 (9)
C7	0.0417 (12)	0.0310 (11)	0.0478 (12)	0.0101 (9)	0.0164 (10)	0.0174 (9)
C8	0.0416 (12)	0.0332 (12)	0.0476 (12)	0.0088 (9)	0.0153 (10)	0.0189 (9)
C9	0.0490 (13)	0.0335 (12)	0.0476 (12)	0.0126 (9)	0.0182 (10)	0.0151 (9)
C10	0.0450 (13)	0.0414 (13)	0.0514 (12)	0.0144 (9)	0.0212 (10)	0.0226 (10)
C11	0.0429 (12)	0.0383 (12)	0.0551 (13)	0.0113 (9)	0.0207 (10)	0.0242 (10)
C12	0.0543 (14)	0.0306 (12)	0.0574 (13)	0.0099 (9)	0.0264 (11)	0.0180 (10)
C13	0.0467 (13)	0.0343 (12)	0.0500 (12)	0.0108 (9)	0.0225 (10)	0.0190 (9)
C14	0.0681 (17)	0.0454 (14)	0.0726 (16)	0.0115 (12)	0.0338 (14)	0.0323 (12)
C15	0.0718 (17)	0.0482 (15)	0.0566 (14)	0.0065 (12)	0.0287 (13)	0.0185 (11)
C16	0.090 (2)	0.0556 (17)	0.0761 (18)	0.0213 (14)	0.0462 (16)	0.0256 (14)
C17	0.0671 (17)	0.0506 (15)	0.0685 (16)	0.0173 (12)	0.0378 (14)	0.0272 (12)
C18	0.0567 (15)	0.0409 (14)	0.0734 (16)	0.0100 (11)	0.0284 (13)	0.0289 (12)
C19	0.0705 (18)	0.0525 (16)	0.103 (2)	0.0137 (13)	0.0413 (17)	0.0444 (15)
C20	0.0461 (13)	0.0355 (12)	0.0484 (12)	0.0045 (9)	0.0173 (10)	0.0203 (10)
C21	0.0550 (15)	0.0514 (15)	0.0626 (15)	0.0112 (11)	0.0156 (12)	0.0305 (12)
C22	0.0561 (16)	0.0651 (19)	0.0697 (17)	0.0000 (13)	0.0007 (13)	0.0373 (15)
C23	0.082 (2)	0.0507 (17)	0.0541 (15)	−0.0072 (14)	−0.0010 (15)	0.0191 (13)
C24	0.0790 (19)	0.0370 (14)	0.0550 (14)	0.0029 (12)	0.0096 (14)	0.0141 (11)
C25	0.0537 (14)	0.0350 (12)	0.0501 (13)	0.0052 (10)	0.0144 (11)	0.0173 (10)
C26	0.0579 (15)	0.0345 (13)	0.0537 (13)	0.0133 (10)	0.0216 (12)	0.0179 (10)
C27	0.0440 (13)	0.0391 (12)	0.0564 (13)	0.0067 (9)	0.0149 (11)	0.0218 (10)
C28	0.0499 (14)	0.0466 (13)	0.0555 (13)	0.0077 (10)	0.0193 (11)	0.0234 (11)
C29	0.0521 (14)	0.0588 (15)	0.0633 (15)	0.0076 (11)	0.0234 (12)	0.0377 (12)
C30	0.0605 (15)	0.0501 (14)	0.0597 (14)	0.0229 (11)	0.0288 (12)	0.0310 (12)
C31	0.096 (2)	0.105 (3)	0.0682 (19)	0.0437 (19)	0.0405 (18)	0.0533 (18)
C32	0.139 (4)	0.131 (3)	0.065 (2)	0.055 (3)	0.026 (2)	0.057 (2)
C33	0.099 (3)	0.100 (3)	0.095 (3)	0.023 (2)	−0.004 (2)	0.060 (2)
C34	0.0605 (18)	0.074 (2)	0.105 (2)	0.0163 (14)	0.0102 (17)	0.0549 (18)
C35	0.0571 (16)	0.0462 (14)	0.0669 (16)	0.0177 (11)	0.0229 (13)	0.0291 (12)

Geometric parameters (Å, º)

O1—C13	1.382 (3)	C15—H15B	0.97
O1—C1	1.384 (2)	C16—H16A	0.96
O2—C26	1.229 (3)	C16—H16B	0.96
O3—C35	1.371 (3)	C16—H16C	0.96
O3—H3A	0.82	C17—H17A	0.96
N1—C26	1.362 (3)	C17—H17B	0.96
N1—C27	1.439 (3)	C17—H17C	0.96
N1—C7	1.491 (3)	C18—C19	1.509 (3)
N2—C28	1.461 (3)	C18—H18A	0.97
N2—C29	1.472 (3)	C18—H18B	0.97
N3—C3	1.372 (3)	C19—H19A	0.96
N3—C15	1.450 (3)	C19—H19B	0.96
N3—H3B	0.86	C19—H19C	0.96
N4—C11	1.385 (3)	C20—C21	1.375 (3)
N4—C18	1.438 (3)	C20—C25	1.379 (3)
N4—H4A	0.86	C21—C22	1.383 (4)
C1—C2	1.375 (3)	C21—H21A	0.93
C1—C6	1.380 (3)	C22—C23	1.376 (4)
C2—C3	1.390 (3)	C22—H22A	0.93
C2—H2B	0.93	C23—C24	1.376 (4)
C3—C4	1.421 (3)	C23—H23A	0.93
C4—C5	1.369 (3)	C24—C25	1.387 (3)
C4—C14	1.506 (3)	C24—H24A	0.93
C5—C6	1.401 (3)	C25—C26	1.466 (3)
C5—H5A	0.93	C27—C28	1.509 (3)
C6—C7	1.512 (3)	C27—H27A	0.97
C7—C8	1.514 (3)	C27—H27B	0.97
C7—C20	1.522 (3)	C28—H28A	0.97
C8—C13	1.379 (3)	C28—H28B	0.97
C8—C9	1.393 (3)	C29—C30	1.498 (3)
C9—C10	1.378 (3)	C29—H29A	0.93
C9—H9A	0.93	C30—C31	1.376 (4)
C10—C11	1.417 (3)	C30—C35	1.390 (3)
C10—C17	1.496 (3)	C31—C32	1.390 (5)
C11—C12	1.377 (3)	C31—H31A	0.93
C12—C13	1.387 (3)	C32—C33	1.360 (5)
C12—H12A	0.93	C32—H32A	0.93
C14—H14A	0.96	C33—C34	1.372 (5)
C14—H14B	0.96	C33—H33A	0.93
C14—H14C	0.96	C34—C35	1.374 (4)
C15—C16	1.501 (4)	C34—H34A	0.93
C15—H15A	0.97		

C13—O1—C1	118.30 (16)	C10—C17—H17A	109.5
C35—O3—H3A	109.5	C10—C17—H17B	109.5
C26—N1—C27	123.37 (19)	H17A—C17—H17B	109.5

C26—N1—C7	113.76 (17)	C10—C17—H17C	109.5
C27—N1—C7	122.35 (16)	H17A—C17—H17C	109.5
C28—N2—C29	112.26 (18)	H17B—C17—H17C	109.5
C3—N3—C15	122.76 (19)	N4—C18—C19	110.8 (2)
C3—N3—H3B	118.6	N4—C18—H18A	109.5
C15—N3—H3B	118.6	C19—C18—H18A	109.5
C11—N4—C18	122.31 (19)	N4—C18—H18B	109.5
C11—N4—H4A	118.8	C19—C18—H18B	109.5
C18—N4—H4A	118.8	H18A—C18—H18B	108.1
C2—C1—C6	122.20 (19)	C18—C19—H19A	109.5
C2—C1—O1	114.70 (18)	C18—C19—H19B	109.5
C6—C1—O1	123.09 (19)	H19A—C19—H19B	109.5
C1—C2—C3	121.2 (2)	C18—C19—H19C	109.5
C1—C2—H2B	119.4	H19A—C19—H19C	109.5
C3—C2—H2B	119.4	H19B—C19—H19C	109.5
N3—C3—C2	121.2 (2)	C21—C20—C25	120.9 (2)
N3—C3—C4	120.58 (19)	C21—C20—C7	128.6 (2)
C2—C3—C4	118.2 (2)	C25—C20—C7	110.5 (2)
C5—C4—C3	118.36 (19)	C20—C21—C22	118.2 (3)
C5—C4—C14	121.3 (2)	C20—C21—H21A	120.9
C3—C4—C14	120.3 (2)	C22—C21—H21A	120.9
C4—C5—C6	124.1 (2)	C23—C22—C21	121.1 (3)
C4—C5—H5A	118.0	C23—C22—H22A	119.5
C6—C5—H5A	118.0	C21—C22—H22A	119.5
C1—C6—C5	116.0 (2)	C22—C23—C24	120.9 (2)
C1—C6—C7	122.45 (18)	C22—C23—H23A	119.5
C5—C6—C7	121.51 (18)	C24—C23—H23A	119.5
N1—C7—C6	111.72 (17)	C23—C24—C25	118.1 (3)
N1—C7—C8	109.84 (17)	C23—C24—H24A	121.0
C6—C7—C8	110.45 (17)	C25—C24—H24A	121.0
N1—C7—C20	99.86 (16)	C20—C25—C24	120.8 (2)
C6—C7—C20	111.95 (17)	C20—C25—C26	109.02 (19)
C8—C7—C20	112.63 (17)	C24—C25—C26	130.1 (2)
C13—C8—C9	116.30 (19)	O2—C26—N1	124.8 (2)
C13—C8—C7	122.44 (19)	O2—C26—C25	128.6 (2)
C9—C8—C7	121.24 (18)	N1—C26—C25	106.6 (2)
C10—C9—C8	123.8 (2)	N1—C27—C28	113.04 (18)
C10—C9—H9A	118.1	N1—C27—H27A	109.0
C8—C9—H9A	118.1	C28—C27—H27A	109.0
C9—C10—C11	118.1 (2)	N1—C27—H27B	109.0
C9—C10—C17	121.2 (2)	C28—C27—H27B	109.0
C11—C10—C17	120.7 (2)	H27A—C27—H27B	107.8
C12—C11—N4	122.2 (2)	N2—C28—C27	110.51 (18)
C12—C11—C10	119.00 (19)	N2—C28—H28A	109.5
N4—C11—C10	118.74 (19)	C27—C28—H28A	109.5
C11—C12—C13	120.7 (2)	N2—C28—H28B	109.5
C11—C12—H12A	119.7	C27—C28—H28B	109.5
C13—C12—H12A	119.7	H28A—C28—H28B	108.1

C8—C13—O1	123.17 (18)	N2—C29—C30	111.97 (19)
C8—C13—C12	122.1 (2)	N2—C29—H29A	124.0
O1—C13—C12	114.72 (18)	C30—C29—H29A	124.0
C4—C14—H14A	109.5	C31—C30—C35	117.9 (3)
C4—C14—H14B	109.5	C31—C30—C29	121.6 (2)
H14A—C14—H14B	109.5	C35—C30—C29	120.4 (2)
C4—C14—H14C	109.5	C30—C31—C32	120.9 (3)
H14A—C14—H14C	109.5	C30—C31—H31A	119.5
H14B—C14—H14C	109.5	C32—C31—H31A	119.5
N3—C15—C16	111.8 (2)	C33—C32—C31	119.8 (3)
N3—C15—H15A	109.2	C33—C32—H32A	120.1
C16—C15—H15A	109.2	C31—C32—H32A	120.1
N3—C15—H15B	109.2	C32—C33—C34	120.5 (3)
C16—C15—H15B	109.2	C32—C33—H33A	119.8
H15A—C15—H15B	107.9	C34—C33—H33A	119.8
C15—C16—H16A	109.5	C33—C34—C35	119.6 (3)
C15—C16—H16B	109.5	C33—C34—H34A	120.2
H16A—C16—H16B	109.5	C35—C34—H34A	120.2
C15—C16—H16C	109.5	O3—C35—C34	118.3 (3)
H16A—C16—H16C	109.5	O3—C35—C30	120.5 (2)
H16B—C16—H16C	109.5	C34—C35—C30	121.2 (3)

C13—O1—C1—C2	177.43 (19)	C9—C8—C13—C12	−0.1 (3)
C13—O1—C1—C6	−2.0 (3)	C7—C8—C13—C12	−178.4 (2)
C6—C1—C2—C3	0.3 (4)	C1—O1—C13—C8	−1.0 (3)
O1—C1—C2—C3	−179.1 (2)	C1—O1—C13—C12	179.64 (19)
C15—N3—C3—C2	1.6 (4)	C11—C12—C13—C8	0.0 (4)
C15—N3—C3—C4	−179.2 (2)	C11—C12—C13—O1	179.4 (2)
C1—C2—C3—N3	177.4 (2)	C3—N3—C15—C16	−175.0 (2)
C1—C2—C3—C4	−1.9 (3)	C11—N4—C18—C19	178.4 (2)
N3—C3—C4—C5	−177.3 (2)	N1—C7—C20—C21	−175.0 (2)
C2—C3—C4—C5	1.9 (3)	C6—C7—C20—C21	−56.6 (3)
N3—C3—C4—C14	1.7 (3)	C8—C7—C20—C21	68.6 (3)
C2—C3—C4—C14	−179.1 (2)	N1—C7—C20—C25	4.0 (2)
C3—C4—C5—C6	−0.5 (3)	C6—C7—C20—C25	122.4 (2)
C14—C4—C5—C6	−179.5 (2)	C8—C7—C20—C25	−112.4 (2)
C2—C1—C6—C5	1.1 (3)	C25—C20—C21—C22	1.1 (3)
O1—C1—C6—C5	−179.5 (2)	C7—C20—C21—C22	180.0 (2)
C2—C1—C6—C7	−175.8 (2)	C20—C21—C22—C23	0.2 (4)
O1—C1—C6—C7	3.6 (3)	C21—C22—C23—C24	−1.0 (4)
C4—C5—C6—C1	−1.0 (3)	C22—C23—C24—C25	0.4 (4)
C4—C5—C6—C7	175.9 (2)	C21—C20—C25—C24	−1.6 (3)
C26—N1—C7—C6	−123.32 (19)	C7—C20—C25—C24	179.3 (2)
C27—N1—C7—C6	64.6 (2)	C21—C20—C25—C26	176.9 (2)
C26—N1—C7—C8	113.8 (2)	C7—C20—C25—C26	−2.2 (3)
C27—N1—C7—C8	−58.3 (2)	C23—C24—C25—C20	0.8 (4)
C26—N1—C7—C20	−4.8 (2)	C23—C24—C25—C26	−177.4 (2)
C27—N1—C7—C20	−176.83 (18)	C27—N1—C26—O2	−5.4 (4)

C1—C6—C7—N1	−124.7 (2)	C7—N1—C26—O2	−177.4 (2)
C5—C6—C7—N1	58.6 (3)	C27—N1—C26—C25	175.75 (19)
C1—C6—C7—C8	−2.1 (3)	C7—N1—C26—C25	3.8 (2)
C5—C6—C7—C8	−178.85 (18)	C20—C25—C26—O2	−179.7 (2)
C1—C6—C7—C20	124.3 (2)	C24—C25—C26—O2	−1.4 (4)
C5—C6—C7—C20	−52.5 (3)	C20—C25—C26—N1	−0.9 (3)
N1—C7—C8—C13	123.0 (2)	C24—C25—C26—N1	177.4 (2)
C6—C7—C8—C13	−0.7 (3)	C26—N1—C27—C28	77.5 (3)
C20—C7—C8—C13	−126.7 (2)	C7—N1—C27—C28	−111.2 (2)
N1—C7—C8—C9	−55.2 (3)	C29—N2—C28—C27	170.9 (2)
C6—C7—C8—C9	−178.86 (19)	N1—C27—C28—N2	176.43 (18)
C20—C7—C8—C9	55.1 (3)	C28—N2—C29—C30	−177.25 (19)
C13—C8—C9—C10	0.1 (3)	N2—C29—C30—C31	−139.0 (3)
C7—C8—C9—C10	178.4 (2)	N2—C29—C30—C35	44.5 (3)
C8—C9—C10—C11	−0.1 (3)	C35—C30—C31—C32	−1.0 (4)
C8—C9—C10—C17	−179.0 (2)	C29—C30—C31—C32	−177.6 (3)
C18—N4—C11—C12	10.5 (4)	C30—C31—C32—C33	0.2 (6)
C18—N4—C11—C10	−170.9 (2)	C31—C32—C33—C34	1.5 (6)
C9—C10—C11—C12	0.0 (3)	C32—C33—C34—C35	−2.3 (5)
C17—C10—C11—C12	178.9 (2)	C33—C34—C35—O3	−179.7 (3)
C9—C10—C11—N4	−178.7 (2)	C33—C34—C35—C30	1.4 (4)
C17—C10—C11—N4	0.2 (3)	C31—C30—C35—O3	−178.7 (2)
N4—C11—C12—C13	178.7 (2)	C29—C30—C35—O3	−2.0 (4)
C10—C11—C12—C13	0.1 (3)	C31—C30—C35—C34	0.2 (4)
C9—C8—C13—O1	−179.5 (2)	C29—C30—C35—C34	176.9 (2)
C7—C8—C13—O1	2.3 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3*A*···N2	0.82	2.01	2.685 (3)	139
N3—H3*B*···O3^{i}	0.86	2.32	3.144 (1)	160
C9—H9*A*···O2^{ii}	0.93	2.59	3.468 (3)	157

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