Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3′,6′-Bis(diethylamino)-2-(2-hydroxyethylamino)spiro[isoindoline-1,9′-xanthen]-3-one

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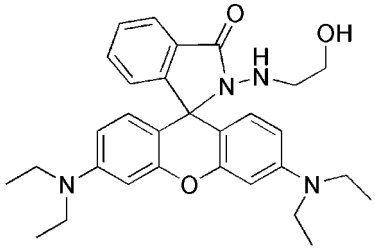
Received 28 May 2008; accepted 6 June 2008

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; disorder in main residue; R factor = 0.051; wR factor = 0.130; data-to-parameter ratio = 12.9.

In the title molecule, $C_{30}H_{36}N_4O_3$, the dihedral angle between the planes of the xanthene and spirolactam rings systems is 88.69 (4)°. Both C atoms of one of the ethyl groups are disordered over two sites with occupancies 0.72 (2)/0.28 (2). The conformation of the molecule may be influenced by two intramolecular hydrogen bonds.

Related literature

For related literature, see: Zhang *et al.* (2007); Wu *et al.* (2007); Bae & Tae 2007).



Experimental

Crystal data

$C_{30}H_{36}N_4O_3$ M_r = 500.63
Monoclinic, $P2_1/c$
a = 12.269 (4) Å
b = 12.203 (4) Å
c = 18.458 (6) Å
β = 108.127 (5)°
V = 2626.4 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 298 (2) K
0.58 × 0.25 × 0.25 mm

Data collection

Bruker SMART APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.849, T_{max} = 0.900
(expected range = 0.924–0.979)
17363 measured reflections
4620 independent reflections
2841 reflections with $I > 2\sigma(I)$
R_{int} = 0.064

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.129
S = 1.00
4620 reflections
359 parameters
13 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3*O*···O2	1.00 (4)	1.79 (4)	2.780 (3)	172 (4)
N4—H1*N*···O2	0.96 (3)	2.45 (2)	2.828 (3)	103 (2)

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

Financial support in part by the Natural Science Foundation of China (20376010 and 20472012) and Shanxi Scholarship Council of China (200310) is gratefully acknowledged.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2636).

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supplementary materials

Acta Cryst. (2008). E**64**, o1252 [**doi:10.1107/S1600536808017108**]

3',6'-Bis(diethylamino)-2-(2-hydroxyethylamino)spiro[isoindoline-1,9'-xanthen]-3-one

M.-Z. Tian and X.-J. Peng

Comment

Rhodamine dyes are molecules used extensively as fluorescent labeling reagents and dye laser sources because of their excellent photophysical properties, such as long absorption and emission wavelengths elongated to visible region, high fluorescence quantum yield, and large absorption coefficient. (Zhang *et al.*, 2007; Wu *et al.*, 2007; Bae & Tae, 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 derivatives, we report here the synthesis and crystal structure of the title compound (I).

As shown in Fig.1, the xanthene ring is close to planar with an r.m.s. deviation of 0.089 (9) Å. The lactam moiety of the molecule is oriented nearly orthogonal to the xanthene moiety *i.e.* the dihedral angle between the planes of the xanthene and the spirolactam rings systems is 88.69 (4)°.

Experimental

Sodium borohydride (15.2 mg, 0.4 mmol) was slowly added to a solution of compound 3',6'-Bis(diethylamino)-2-(2-oxoethylideneamino)spiro [isoindoline-1,9'-xanthen]-3-one (150 mg, 0.3 mmol) in ethanol (20 ml). The reaction mixture was stirred for 2 h at room temperature and solvent was totally removed under reduced pressure. The crude product was dissolved in CH_2Cl_2 (20 ml) and 3 ml of an aqueous solution of K_2CO_3 was added. The organic layer was dried over $MgSO_4$. After filtration, the solvent was removed under reduced pressure. The residue was placed on a silica gel column (200–300 mesh). The column was eluted with a mixture (2:1, *v/v*) of petroleum spirit/ethyl acetate, to give 136 mg of the title compound (90%). Crystals were grown by dissolving the compound in CH_2Cl_2 and slowly diffusing n-hexane into the solution.

Refinement

H atoms bonded to Catoms were positioned geometrically (C—H = 0.93–0.97 Å) and refined as riding, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). The positional parameters of the H atoms bonded to N and O were refined independently with U_{iso}(H) = 1.5U_{eq}(N,*O*).

Figures

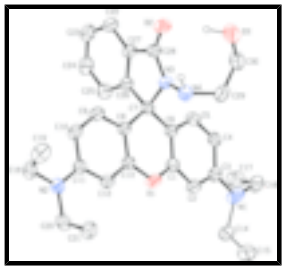


Fig. 1. The molecular structure of (I), showing the atom-numbering scheme and displacement ellipsoids at the 30% probability level. H atoms bonded to C atoms have been omitted and the disorder is not shown.

3',6'-Bis(diethylamino)-2-(2-hydroxyethylamino)spiro[isoindoline-1,9'- xanthen]-3-one

Crystal data

$C_{30}H_{36}N_4O_3$	F_{000} = 1072
M_r = 500.63	D_x = 1.266 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 3630 reflections
a = 12.269 (4) Å	θ = 2.0–25.0º
b = 12.203 (4) Å	μ = 0.08 mm^{-1}
c = 18.458 (6) Å	T = 298 (2) K
β = 108.127 (5)º	Block, white
V = 2626.4 (15) Å^3	0.58 × 0.25 × 0.25 mm
Z = 4	

Data collection

Bruker SMART APEXII diffractometer	4620 independent reflections
Radiation source: fine-focus sealed tube	2841 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.064
T = 298(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 2.0º
Absorption correction: multi-scan (SADABS; Bruker, 2005)	h = −14→14
T_{min} = 0.849, T_{max} = 0.900	k = −14→14
17363 measured reflections	l = −21→21

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.050	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.129	$w = 1/[\sigma^2(F_o^2) + (0.0755P)^2]$

	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{max} = 0.002$
4620 reflections	$\Delta\rho_{max} = 0.21$ e Å^{-3}
359 parameters	$\Delta\rho_{min} = -0.20$ e Å^{-3}
13 restraints	Extinction correction: SHELXL97 (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.026 (4)

Special details

Experimental. ^{1}H NMR ($CDCl_3$, 400MHz, Me_4Si): δ 7.91 (d, 1H, *J*=6.4 Hz, C_6H_4), 7.52-7.47 (m, 2H, C_6H_4), 7.15 (d, 1H, *J*= 6.4 Hz), 6.41 (m, 4H, Xanthene-H), 6.26 (dd, 2H, *J*= 8.8 Hz, *J*= 2.4 Hz, Xanthene-H), 4.65 (t, 1H, *J*= 7.2 Hz, NH), 4.45 (t, 1H, *J*= 6.0 Hz, OH), 3.36-3.31 (m, 10H, CH_2O, CH_2), 2.46-2.45 (m, 2H, CH_2N), 1.16 (t, 12H, *J*=6.8 Hz, CH_3); ^{13}C NMR ($CDCl_3$, 100MHz, Me_4Si): δ 168.31, 164.31, 153.97, 151.59, 149.05, 133.17, 129.99, 128.51, 123.08, 124.20, 107.97, 105.29, 97.98, 66.39, 58.76, 52.83, 44.51, 12.75. HRMS(ESI): calcd for $C_{30}H_{36}N_4O_3$ $[M+Na]^+$ 523.2685; found 523.2671.

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}	Occ. (<1)
O1	0.02779 (11)	0.63551 (12)	0.15665 (9)	0.0590 (5)	
O2	−0.40178 (12)	0.91274 (15)	0.13067 (9)	0.0670 (5)	
O3	−0.34132 (15)	0.9162 (2)	0.28884 (13)	0.0941 (7)	
H3O	−0.370 (3)	0.915 (3)	0.232 (2)	0.141*	
N1	0.31949 (16)	0.86643 (19)	0.30367 (13)	0.0770 (7)	
N2	−0.19898 (16)	0.38340 (17)	−0.02009 (11)	0.0620 (5)	
N3	−0.22666 (13)	0.83188 (15)	0.15192 (9)	0.0443 (4)	
N4	−0.23247 (17)	0.76085 (17)	0.20976 (11)	0.0570 (5)	
H1N	−0.313 (2)	0.749 (2)	0.2013 (14)	0.085*	
C1	0.05554 (16)	0.74041 (19)	0.18219 (11)	0.0459 (5)	
C2	0.16682 (17)	0.7528 (2)	0.22812 (12)	0.0551 (6)	
H2A	0.2155	0.6925	0.2386	0.066*	
C3	0.20776 (17)	0.8541 (2)	0.25922 (13)	0.0525 (6)	
C4	0.13007 (17)	0.94070 (19)	0.24316 (12)	0.0488 (6)	
H4A	0.1530	1.0093	0.2643	0.059*	
C5	0.02040 (17)	0.92561 (18)	0.19652 (12)	0.0468 (5)	
H5A	−0.0292	0.9853	0.1863	0.056*	
C6	−0.02056 (15)	0.82580 (18)	0.16369 (11)	0.0407 (5)	

C7	−0.14135 (15)	0.81057 (17)	0.11196 (11)	0.0417 (5)	
C8	−0.15672 (16)	0.69727 (18)	0.07841 (11)	0.0439 (5)	
C9	−0.25529 (17)	0.6667 (2)	0.02086 (13)	0.0570 (6)	
H9A	−0.3130	0.7186	0.0030	0.068*	
C10	−0.27173 (19)	0.5653 (2)	−0.01066 (13)	0.0593 (6)	
H10A	−0.3398	0.5491	−0.0487	0.071*	
C11	−0.18663 (17)	0.48494 (19)	0.01373 (12)	0.0501 (6)	
C12	−0.08854 (17)	0.51411 (19)	0.07132 (12)	0.0494 (6)	
H12A	−0.0307	0.4626	0.0898	0.059*	
C13	−0.07482 (16)	0.61794 (18)	0.10178 (11)	0.0436 (5)	
C14	0.4083 (4)	0.7842 (6)	0.2970 (4)	0.062 (2)	0.720 (17)
H14A	0.3815	0.7481	0.2478	0.074*	0.720 (17)
H14B	0.4792	0.8221	0.3002	0.074*	0.720 (17)
C15	0.4301 (7)	0.7010 (7)	0.3586 (4)	0.099 (2)	0.720 (17)
H15A	0.4863	0.6496	0.3533	0.149*	0.720 (17)
H15B	0.3601	0.6631	0.3550	0.149*	0.720 (17)
H15C	0.4580	0.7367	0.4072	0.149*	0.720 (17)
C14A	0.3796 (13)	0.7676 (16)	0.3463 (9)	0.074 (7)	0.280 (17)
H14C	0.4289	0.7869	0.3969	0.088*	0.280 (17)
H14D	0.3254	0.7120	0.3505	0.088*	0.280 (17)
C15A	0.4469 (17)	0.731 (2)	0.2969 (13)	0.111 (7)	0.280 (17)
H15D	0.4904	0.6668	0.3187	0.166*	0.280 (17)
H15E	0.4983	0.7881	0.2928	0.166*	0.280 (17)
H15F	0.3959	0.7135	0.2472	0.166*	0.280 (17)
C16	0.3656 (2)	0.9732 (2)	0.33183 (16)	0.0739 (8)	
H16A	0.4332	0.9633	0.3757	0.089*	
H16B	0.3092	1.0128	0.3485	0.089*	
C17	0.3969 (2)	1.0400 (3)	0.27363 (18)	0.0852 (9)	
H17A	0.4269	1.1094	0.2953	0.128*	
H17B	0.3299	1.0517	0.2305	0.128*	
H17C	0.4539	1.0020	0.2576	0.128*	
C18	−0.3044 (2)	0.3522 (2)	−0.07672 (14)	0.0690 (7)	
H18A	−0.2890	0.2907	−0.1054	0.083*	
H18B	−0.3301	0.4127	−0.1120	0.083*	
C19	−0.3997 (2)	0.3211 (3)	−0.04619 (17)	0.0877 (9)	
H19A	−0.4665	0.3023	−0.0877	0.132*	
H19B	−0.4170	0.3817	−0.0185	0.132*	
H19C	−0.3766	0.2592	−0.0127	0.132*	
C20	−0.1110 (2)	0.2997 (2)	0.00744 (14)	0.0665 (7)	
H20A	−0.0363	0.3339	0.0183	0.080*	
H20B	−0.1183	0.2465	−0.0328	0.080*	
C21	−0.1165 (2)	0.2407 (2)	0.07736 (15)	0.0744 (8)	
H21A	−0.0561	0.1874	0.0921	0.112*	
H21B	−0.1893	0.2047	0.0668	0.112*	
H21C	−0.1075	0.2924	0.1180	0.112*	
C22	−0.34216 (19)	1.0120 (2)	−0.00789 (14)	0.0593 (6)	
H22A	−0.4118	1.0420	−0.0078	0.071*	
C23	−0.2949 (2)	1.0373 (2)	−0.06426 (14)	0.0654 (7)	
H23A	−0.3340	1.0832	−0.1041	0.078*	

C24	−0.1899 (2)	0.9951 (2)	−0.06186 (14)	0.0692 (7)
H24A	−0.1574	1.0154	−0.0991	0.083*
C25	−0.13217 (19)	0.9237 (2)	−0.00578 (13)	0.0571 (6)
H25A	−0.0617	0.8949	−0.0051	0.068*
C26	−0.18049 (16)	0.89571 (18)	0.04935 (12)	0.0455 (5)
C27	−0.28307 (16)	0.94066 (18)	0.04841 (12)	0.0482 (6)
C28	−0.31390 (17)	0.89661 (19)	0.11342 (12)	0.0499 (6)
C29	−0.1807 (2)	0.8051 (3)	0.28589 (13)	0.0694 (8)
H29A	−0.0986	0.8101	0.2953	0.083*
H29B	−0.1933	0.7534	0.3224	0.083*
C30	−0.2233 (2)	0.9153 (3)	0.30112 (16)	0.0804 (9)
H30A	−0.1842	0.9361	0.3535	0.097*
H30B	−0.2048	0.9693	0.2682	0.097*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0457 (8)	0.0423 (10)	0.0714 (10)	0.0041 (7)	−0.0072 (7)	−0.0086 (8)
O2	0.0416 (8)	0.0841 (14)	0.0751 (11)	0.0098 (8)	0.0178 (8)	0.0037 (9)
O3	0.0625 (11)	0.143 (2)	0.0822 (13)	0.0030 (11)	0.0306 (10)	−0.0282 (13)
N1	0.0511 (11)	0.0595 (16)	0.0937 (16)	0.0007 (10)	−0.0163 (11)	−0.0167 (13)
N2	0.0701 (12)	0.0476 (13)	0.0588 (13)	−0.0039 (10)	0.0065 (10)	−0.0138 (10)
N3	0.0412 (9)	0.0439 (11)	0.0479 (10)	0.0031 (8)	0.0141 (7)	0.0043 (9)
N4	0.0620 (11)	0.0576 (14)	0.0535 (12)	0.0019 (10)	0.0211 (10)	0.0056 (10)
C1	0.0424 (11)	0.0430 (15)	0.0490 (13)	−0.0023 (10)	0.0093 (9)	−0.0057 (10)
C2	0.0467 (12)	0.0491 (16)	0.0587 (14)	0.0057 (11)	0.0009 (10)	−0.0040 (12)
C3	0.0426 (11)	0.0570 (17)	0.0505 (13)	−0.0034 (11)	0.0039 (9)	−0.0047 (11)
C4	0.0479 (12)	0.0478 (15)	0.0502 (13)	−0.0037 (10)	0.0146 (10)	−0.0092 (11)
C5	0.0431 (11)	0.0436 (15)	0.0547 (13)	0.0025 (10)	0.0167 (10)	−0.0038 (11)
C6	0.0376 (10)	0.0411 (14)	0.0427 (11)	0.0011 (9)	0.0115 (8)	0.0000 (10)
C7	0.0368 (10)	0.0389 (14)	0.0484 (12)	−0.0003 (9)	0.0117 (9)	−0.0015 (10)
C8	0.0395 (10)	0.0429 (14)	0.0467 (12)	0.0005 (10)	0.0097 (9)	−0.0005 (10)
C9	0.0465 (12)	0.0463 (16)	0.0650 (15)	0.0033 (11)	−0.0019 (10)	−0.0006 (12)
C10	0.0540 (13)	0.0500 (16)	0.0581 (15)	−0.0037 (12)	−0.0055 (11)	−0.0046 (12)
C11	0.0545 (13)	0.0475 (15)	0.0454 (13)	−0.0040 (11)	0.0115 (10)	−0.0019 (11)
C12	0.0487 (12)	0.0407 (15)	0.0538 (14)	0.0038 (10)	0.0086 (10)	−0.0014 (11)
C13	0.0382 (10)	0.0451 (14)	0.0430 (12)	−0.0011 (10)	0.0061 (9)	−0.0005 (10)
C14	0.039 (2)	0.073 (4)	0.067 (3)	−0.001 (2)	0.007 (2)	−0.008 (3)
C15	0.116 (4)	0.091 (5)	0.083 (4)	0.042 (4)	0.021 (3)	0.013 (3)
C14A	0.058 (8)	0.085 (14)	0.068 (9)	−0.009 (8)	0.006 (6)	−0.020 (7)
C15A	0.089 (11)	0.114 (16)	0.137 (16)	0.022 (9)	0.046 (10)	−0.030 (12)
C16	0.0539 (13)	0.074 (2)	0.0739 (18)	−0.0028 (13)	−0.0099 (13)	−0.0210 (16)
C17	0.0611 (16)	0.084 (2)	0.109 (2)	−0.0023 (15)	0.0243 (16)	−0.0150 (19)
C18	0.0865 (17)	0.0548 (18)	0.0527 (15)	−0.0111 (14)	0.0029 (13)	−0.0077 (12)
C19	0.0829 (18)	0.075 (2)	0.092 (2)	−0.0107 (16)	0.0089 (16)	0.0116 (17)
C20	0.0777 (16)	0.0508 (17)	0.0700 (17)	−0.0048 (13)	0.0216 (13)	−0.0173 (13)
C21	0.0750 (17)	0.071 (2)	0.0689 (17)	−0.0050 (14)	0.0101 (13)	−0.0033 (15)
C22	0.0542 (13)	0.0487 (16)	0.0643 (16)	0.0053 (11)	0.0026 (12)	0.0008 (13)

C23	0.0765 (17)	0.0556 (17)	0.0541 (15)	−0.0006 (13)	0.0058 (13)	0.0085 (13)
C24	0.0820 (17)	0.071 (2)	0.0526 (15)	−0.0097 (15)	0.0179 (13)	0.0046 (14)
C25	0.0563 (13)	0.0583 (17)	0.0585 (15)	0.0003 (12)	0.0206 (11)	0.0047 (13)
C26	0.0426 (11)	0.0405 (14)	0.0485 (13)	−0.0011 (9)	0.0069 (9)	−0.0037 (10)
C27	0.0421 (11)	0.0428 (14)	0.0529 (14)	−0.0006 (10)	0.0048 (9)	0.0013 (11)
C28	0.0363 (11)	0.0513 (15)	0.0580 (14)	0.0023 (10)	0.0086 (10)	−0.0027 (11)
C29	0.0540 (13)	0.103 (2)	0.0488 (15)	0.0018 (14)	0.0126 (11)	0.0089 (15)
C30	0.0612 (15)	0.114 (3)	0.0668 (17)	−0.0129 (16)	0.0209 (13)	−0.0303 (17)

Geometric parameters (Å, °)

O1—C13	1.365 (2)	C15—H15B	0.9600
O1—C1	1.370 (3)	C15—H15C	0.9600
O2—C28	1.231 (2)	C14A—C15A	1.48 (4)
O3—C30	1.394 (3)	C14A—H14C	0.9700
O3—H3O	0.99 (4)	C14A—H14D	0.9700
N1—C3	1.369 (3)	C15A—H15D	0.9600
N1—C16	1.450 (3)	C15A—H15E	0.9600
N1—C14A	1.50 (2)	C15A—H15F	0.9600
N1—C14	1.514 (8)	C16—C17	1.491 (4)
N2—C11	1.374 (3)	C16—H16A	0.9700
N2—C18	1.438 (3)	C16—H16B	0.9700
N2—C20	1.457 (3)	C17—H17A	0.9600
N3—C28	1.342 (3)	C17—H17B	0.9600
N3—N4	1.394 (2)	C17—H17C	0.9600
N3—C7	1.479 (2)	C18—C19	1.496 (4)
N4—C29	1.454 (3)	C18—H18A	0.9700
N4—H1N	0.96 (3)	C18—H18B	0.9700
C1—C6	1.370 (3)	C19—H19A	0.9600
C1—C2	1.373 (3)	C19—H19B	0.9600
C2—C3	1.389 (3)	C19—H19C	0.9600
C2—H2A	0.9300	C20—C21	1.497 (4)
C3—C4	1.392 (3)	C20—H20A	0.9700
C4—C5	1.365 (3)	C20—H20B	0.9700
C4—H4A	0.9300	C21—H21A	0.9600
C5—C6	1.383 (3)	C21—H21B	0.9600
C5—H5A	0.9300	C21—H21C	0.9600
C6—C7	1.504 (3)	C22—C23	1.375 (3)
C7—C8	1.503 (3)	C22—C27	1.376 (3)
C7—C26	1.517 (3)	C22—H22A	0.9300
C8—C13	1.365 (3)	C23—C24	1.375 (3)
C8—C9	1.389 (3)	C23—H23A	0.9300
C9—C10	1.355 (3)	C24—C25	1.370 (3)
C9—H9A	0.9300	C24—H24A	0.9300
C10—C11	1.401 (3)	C25—C26	1.371 (3)
C10—H10A	0.9300	C25—H25A	0.9300
C11—C12	1.382 (3)	C26—C27	1.368 (3)
C12—C13	1.375 (3)	C27—C28	1.468 (3)
C12—H12A	0.9300	C29—C30	1.501 (4)

C14—C15	1.485 (13)	C29—H29A	0.9700
C14—H14A	0.9700	C29—H29B	0.9700
C14—H14B	0.9700	C30—H30A	0.9700
C15—H15A	0.9600	C30—H30B	0.9700
C13—O1—C1	118.37 (16)	H14C—C14A—H14D	109.3
C30—O3—H3O	101 (2)	C14A—C15A—H15D	109.5
C3—N1—C16	121.3 (2)	C14A—C15A—H15E	109.5
C3—N1—C14A	117.6 (5)	H15D—C15A—H15E	109.5
C16—N1—C14A	117.4 (4)	C14A—C15A—H15F	109.5
C3—N1—C14	119.3 (2)	H15D—C15A—H15F	109.5
C16—N1—C14	114.5 (2)	H15E—C15A—H15F	109.5
C14A—N1—C14	42.5 (6)	N1—C16—C17	113.1 (2)
C11—N2—C18	121.1 (2)	N1—C16—H16A	109.0
C11—N2—C20	120.56 (19)	C17—C16—H16A	109.0
C18—N2—C20	117.9 (2)	N1—C16—H16B	109.0
C28—N3—N4	123.30 (17)	C17—C16—H16B	109.0
C28—N3—C7	114.26 (17)	H16A—C16—H16B	107.8
N4—N3—C7	119.03 (16)	C16—C17—H17A	109.5
N3—N4—C29	113.5 (2)	C16—C17—H17B	109.5
N3—N4—H1N	105.4 (16)	H17A—C17—H17B	109.5
C29—N4—H1N	109.4 (15)	C16—C17—H17C	109.5
C6—C1—O1	123.23 (17)	H17A—C17—H17C	109.5
C6—C1—C2	122.7 (2)	H17B—C17—H17C	109.5
O1—C1—C2	114.11 (19)	N2—C18—C19	115.1 (2)
C1—C2—C3	121.0 (2)	N2—C18—H18A	108.5
C1—C2—H2A	119.5	C19—C18—H18A	108.5
C3—C2—H2A	119.5	N2—C18—H18B	108.5
N1—C3—C2	120.7 (2)	C19—C18—H18B	108.5
N1—C3—C4	122.4 (2)	H18A—C18—H18B	107.5
C2—C3—C4	116.86 (19)	C18—C19—H19A	109.5
C5—C4—C3	120.5 (2)	C18—C19—H19B	109.5
C5—C4—H4A	119.8	H19A—C19—H19B	109.5
C3—C4—H4A	119.8	C18—C19—H19C	109.5
C4—C5—C6	123.2 (2)	H19A—C19—H19C	109.5
C4—C5—H5A	118.4	H19B—C19—H19C	109.5
C6—C5—H5A	118.4	N2—C20—C21	114.2 (2)
C1—C6—C5	115.73 (18)	N2—C20—H20A	108.7
C1—C6—C7	121.66 (19)	C21—C20—H20A	108.7
C5—C6—C7	122.61 (18)	N2—C20—H20B	108.7
N3—C7—C8	110.47 (16)	C21—C20—H20B	108.7
N3—C7—C6	111.84 (16)	H20A—C20—H20B	107.6
C8—C7—C6	110.36 (16)	C20—C21—H21A	109.5
N3—C7—C26	98.86 (15)	C20—C21—H21B	109.5
C8—C7—C26	110.30 (17)	H21A—C21—H21B	109.5
C6—C7—C26	114.52 (17)	C20—C21—H21C	109.5
C13—C8—C9	115.6 (2)	H21A—C21—H21C	109.5
C13—C8—C7	122.46 (18)	H21B—C21—H21C	109.5
C9—C8—C7	121.98 (19)	C23—C22—C27	117.8 (2)
C10—C9—C8	123.4 (2)	C23—C22—H22A	121.1

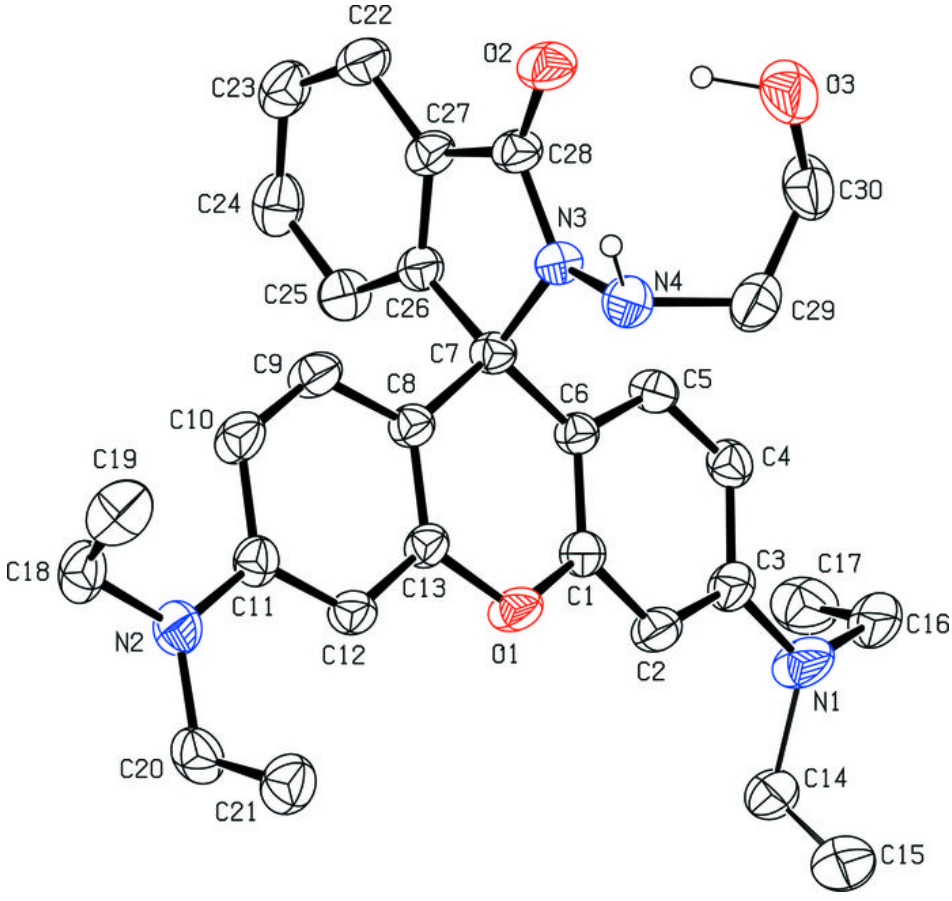
C10—C9—H9A	118.3	C27—C22—H22A	121.1
C8—C9—H9A	118.3	C24—C23—C22	120.2 (2)
C9—C10—C11	120.3 (2)	C24—C23—H23A	119.9
C9—C10—H10A	119.8	C22—C23—H23A	119.9
C11—C10—H10A	119.8	C25—C24—C23	121.5 (2)
N2—C11—C12	121.9 (2)	C25—C24—H24A	119.3
N2—C11—C10	121.3 (2)	C23—C24—H24A	119.3
C12—C11—C10	116.8 (2)	C24—C25—C26	118.6 (2)
C13—C12—C11	121.2 (2)	C24—C25—H25A	120.7
C13—C12—H12A	119.4	C26—C25—H25A	120.7
C11—C12—H12A	119.4	C27—C26—C25	119.9 (2)
O1—C13—C8	122.78 (19)	C27—C26—C7	110.87 (18)
O1—C13—C12	114.55 (18)	C25—C26—C7	128.97 (19)
C8—C13—C12	122.67 (19)	C26—C27—C22	122.0 (2)
C15—C14—N1	110.7 (7)	C26—C27—C28	108.24 (18)
C15—C14—H14A	109.5	C22—C27—C28	129.8 (2)
N1—C14—H14A	109.5	O2—C28—N3	125.1 (2)
C15—C14—H14B	109.5	O2—C28—C27	128.5 (2)
N1—C14—H14B	109.5	N3—C28—C27	106.41 (17)
H14A—C14—H14B	108.1	N4—C29—C30	116.2 (2)
C14—C15—H15A	109.5	N4—C29—H29A	108.2
C14—C15—H15B	109.5	C30—C29—H29A	108.2
H15A—C15—H15B	109.5	N4—C29—H29B	108.2
C14—C15—H15C	109.5	C30—C29—H29B	108.2
H15A—C15—H15C	109.5	H29A—C29—H29B	107.4
H15B—C15—H15C	109.5	O3—C30—C29	112.4 (2)
C15A—C14A—N1	101.4 (18)	O3—C30—H30A	109.1
C15A—C14A—H14C	111.5	C29—C30—H30A	109.1
N1—C14A—H14C	111.5	O3—C30—H30B	109.1
C15A—C14A—H14D	111.5	C29—C30—H30B	109.1
N1—C14A—H14D	111.5	H30A—C30—H30B	107.9
C28—N3—N4—C29	−98.3 (2)	N2—C11—C12—C13	176.4 (2)
C7—N3—N4—C29	103.7 (2)	C10—C11—C12—C13	−1.3 (3)
C13—O1—C1—C6	9.6 (3)	C1—O1—C13—C8	−8.6 (3)
C13—O1—C1—C2	−170.31 (18)	C1—O1—C13—C12	170.74 (18)
C6—C1—C2—C3	0.7 (4)	C9—C8—C13—O1	178.64 (18)
O1—C1—C2—C3	−179.3 (2)	C7—C8—C13—O1	−0.8 (3)
C16—N1—C3—C2	175.4 (2)	C9—C8—C13—C12	−0.6 (3)
C14A—N1—C3—C2	−27.0 (8)	C7—C8—C13—C12	179.92 (19)
C14—N1—C3—C2	21.6 (5)	C11—C12—C13—O1	−178.19 (19)
C16—N1—C3—C4	−4.9 (4)	C11—C12—C13—C8	1.1 (3)
C14A—N1—C3—C4	152.7 (8)	C3—N1—C14—C15	−98.4 (4)
C14—N1—C3—C4	−158.7 (4)	C16—N1—C14—C15	106.1 (4)
C1—C2—C3—N1	−178.6 (2)	C14A—N1—C14—C15	1.5 (7)
C1—C2—C3—C4	1.6 (3)	C3—N1—C14A—C15A	100.1 (9)
N1—C3—C4—C5	177.9 (2)	C16—N1—C14A—C15A	−101.4 (9)
C2—C3—C4—C5	−2.4 (3)	C14—N1—C14A—C15A	−4.3 (8)
C3—C4—C5—C6	0.9 (3)	C3—N1—C16—C17	−79.7 (3)
O1—C1—C6—C5	177.82 (18)	C14A—N1—C16—C17	122.7 (8)

C2—C1—C6—C5	−2.2 (3)	C14—N1—C16—C17	75.3 (4)
O1—C1—C6—C7	−1.2 (3)	C11—N2—C18—C19	−77.1 (3)
C2—C1—C6—C7	178.74 (19)	C20—N2—C18—C19	95.2 (3)
C4—C5—C6—C1	1.5 (3)	C11—N2—C20—C21	79.8 (3)
C4—C5—C6—C7	−179.54 (19)	C18—N2—C20—C21	−92.5 (3)
C28—N3—C7—C8	−103.7 (2)	C27—C22—C23—C24	−2.3 (4)
N4—N3—C7—C8	56.2 (2)	C22—C23—C24—C25	2.8 (4)
C28—N3—C7—C6	132.94 (19)	C23—C24—C25—C26	−0.9 (4)
N4—N3—C7—C6	−67.2 (2)	C24—C25—C26—C27	−1.4 (4)
C28—N3—C7—C26	11.9 (2)	C24—C25—C26—C7	172.8 (2)
N4—N3—C7—C26	171.83 (17)	N3—C7—C26—C27	−9.7 (2)
C1—C6—C7—N3	116.1 (2)	C8—C7—C26—C27	106.1 (2)
C5—C6—C7—N3	−62.9 (3)	C6—C7—C26—C27	−128.69 (19)
C1—C6—C7—C8	−7.3 (3)	N3—C7—C26—C25	175.7 (2)
C5—C6—C7—C8	173.74 (18)	C8—C7—C26—C25	−68.6 (3)
C1—C6—C7—C26	−132.5 (2)	C6—C7—C26—C25	56.6 (3)
C5—C6—C7—C26	48.6 (3)	C25—C26—C27—C22	1.8 (3)
N3—C7—C8—C13	−115.9 (2)	C7—C26—C27—C22	−173.42 (19)
C6—C7—C8—C13	8.3 (3)	C25—C26—C27—C28	−179.9 (2)
C26—C7—C8—C13	135.9 (2)	C7—C26—C27—C28	4.9 (2)
N3—C7—C8—C9	64.7 (3)	C23—C22—C27—C26	0.1 (3)
C6—C7—C8—C9	−171.09 (18)	C23—C22—C27—C28	−177.8 (2)
C26—C7—C8—C9	−43.6 (3)	N4—N3—C28—O2	10.5 (3)
C13—C8—C9—C10	0.5 (3)	C7—N3—C28—O2	169.5 (2)
C7—C8—C9—C10	179.9 (2)	N4—N3—C28—C27	−168.71 (18)
C8—C9—C10—C11	−0.8 (4)	C7—N3—C28—C27	−9.8 (2)
C18—N2—C11—C12	176.2 (2)	C26—C27—C28—O2	−176.5 (2)
C20—N2—C11—C12	4.1 (3)	C22—C27—C28—O2	1.7 (4)
C18—N2—C11—C10	−6.1 (3)	C26—C27—C28—N3	2.8 (2)
C20—N2—C11—C10	−178.3 (2)	C22—C27—C28—N3	−179.1 (2)
C9—C10—C11—N2	−176.6 (2)	N3—N4—C29—C30	53.9 (3)
C9—C10—C11—C12	1.2 (4)	N4—C29—C30—O3	57.8 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3O···O2	1.00 (4)	1.79 (4)	2.780 (3)	172 (4)
N4—H1N···O2	0.96 (3)	2.45 (2)	2.828 (3)	103 (2)

Fig. 1


C22
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C3
C17
C16
N2
C12
O1
C2
N1
C20
C14
C21
C15