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

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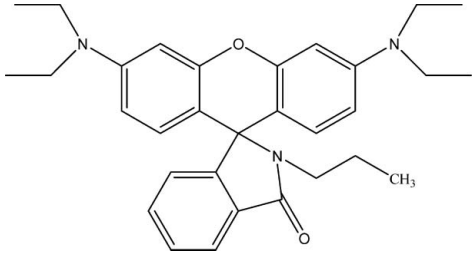
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.004 Å; R factor = 0.061; wR factor = 0.201; data-to-parameter ratio = 14.2.

In the crystal structure of the title compound, $C_{31}H_{37}N_3O_2$, the dihedral angle between the planes of the xanthene ring system and the spirolactam ring is 90.3°.

Related literature

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



Experimental

Crystal data

$C_{31}H_{37}N_3O_2$
M_r = 483.64
Triclinic, $P\overline{1}$
a = 11.1697 (4) Å
b = 11.8605 (4) Å
c = 12.4512 (4) Å
α = 82.838 (3)°
β = 64.605 (2)°
γ = 65.588 (2)°
V = 1354.13 (8) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 273 (2) K
0.55 × 0.40 × 0.20 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.960, T_{max} = 0.985
9526 measured reflections
4699 independent reflections
2963 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.201
S = 1.07
4699 reflections
330 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); 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: NC2050).

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supplementary materials

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

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Comment

Rhodamine dyes are widely used as fluorescent labels (De Silva *et al.*, 1997) but only a few of such compounds were characterized by single-crystal X-ray diffraction (Kwon *et al.*, 2005). However, for the understanding of their photophysical and photochemical properties, information on their molecular and crystal structures are needed. As part of our ongoing work on rhodamine dyes, we report here the synthesis and crystal structure of the title compound 3',6'-Bis(diethylamino)-2-propyl-spiro[isoindoline-1,9'-[xanthen]-3-one (I).

As similar compounds the main skeleton of the molecule is built up of an xanthene ring which is connected to an spirolactam-ring (Kwon *et al.*, 2005). The lactam moiety is oriented nearly orthogonal to the xanthene moiety. The dihedral angle between the planes of the xanthene ring and the spirolactam-ring amount to 90.3°.

Experimental

Rhodamine B(1 g, 2.26 mmol) and 1-aminopropane (0.186 ml, 2.26 mmol) were refluxed in 20 ml of methanol for about 3 h until the color changes into light yellow. The crude product were purified by column chromatography (eluation with MeOH/CH_2Cl_2 = 100:1, *v/v*). After removal of the solvent in vacuum the solid were dried in vacuum to afford the title compound in 53% yield. Single crystals suitable for X-ray measurements were obtained from a hexane/dichloromethane (1:1, *v/v*) solution by slow evaporation of the solvent at room temperature.

Refinement

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

Figures

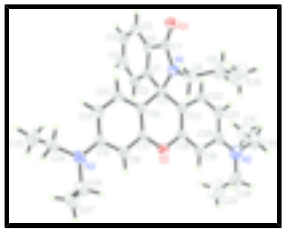


Fig. 1. Crystal structure of compound I, with labelling and displacement ellipsoids drawn at the 30% probability level.

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

Crystal data

$C_{31}H_{37}N_3O_2$	$Z = 2$
$M_r = 483.64$	$F_{000} = 520$
Triclinic, $P\overline{1}$	$D_x = 1.186$ Mg m^{-3}
Hall symbol: -P 1	Melting point: 482 K
$a = 11.1697$ (4) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 11.8605$ (4) Å	Cell parameters from 2231 reflections
$c = 12.4512$ (4) Å	$\theta = 3.5$–22.9º
$\alpha = 82.838$ (3)º	$\mu = 0.07$ mm^{-1}
$\beta = 64.605$ (2)º	$T = 273$ (2) K
$\gamma = 65.588$ (2)º	Block, white
$V = 1354.13$ (8) Å^3	0.55 × 0.40 × 0.20 mm

Data collection

Bruker APEXII CCD area-detector diffractometer	4699 independent reflections
Radiation source: fine-focus sealed tube	2963 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.027$
$T = 273(2)$ K	$\theta_{max} = 25.0$º
φ and ω scans	$\theta_{min} = 2.7$º
Absorption correction: multi-scan (SADABS; Bruker, 1997)	$h = -13 \rightarrow 13$
$T_{min} = 0.960$, $T_{max} = 0.985$	$k = -13 \rightarrow 14$
9526 measured reflections	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.1086P)^2 + 0.0965P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.061$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.201$	$\Delta\rho_{max} = 0.32$ e Å^{-3}
$S = 1.07$	$\Delta\rho_{min} = -0.24$ e Å^{-3}
4699 reflections	Extinction correction: none
330 parameters	

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 $F^2 > 2\text{sigma}(F^2)$ 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}
O2	0.6833 (2)	0.94710 (14)	0.39573 (15)	0.0663 (5)
C23	0.6694 (2)	1.1572 (2)	0.3635 (2)	0.0504 (6)
C12	0.6414 (2)	1.0462 (2)	0.2257 (2)	0.0510 (6)
C17	0.6604 (2)	0.9467 (2)	0.2956 (2)	0.0519 (6)
N1	0.5045 (2)	1.27294 (17)	0.26636 (17)	0.0539 (5)
C16	0.6599 (3)	0.8367 (2)	0.2691 (2)	0.0578 (6)
H16A	0.6742	0.7713	0.3182	0.069*
O1	0.4211 (2)	1.44106 (16)	0.16568 (17)	0.0752 (6)
C15	0.6385 (2)	0.8222 (2)	0.1706 (2)	0.0572 (6)
C18	0.6879 (3)	1.0524 (2)	0.4273 (2)	0.0534 (6)
C14	0.6199 (3)	0.9239 (2)	0.0986 (3)	0.0647 (7)
H14A	0.6069	0.9181	0.0309	0.078*
C11	0.6423 (2)	1.16741 (19)	0.2536 (2)	0.0507 (6)
C19	0.7147 (3)	1.0421 (2)	0.5268 (2)	0.0620 (7)
H19A	0.7286	0.9685	0.5654	0.074*
C1	0.7490 (3)	1.2028 (2)	0.1450 (2)	0.0527 (6)
C7	0.5201 (3)	1.3512 (2)	0.1766 (2)	0.0556 (6)
C13	0.6208 (3)	1.0308 (2)	0.1271 (2)	0.0619 (7)
H13A	0.6069	1.0965	0.0782	0.074*
C22	0.6769 (3)	1.2555 (2)	0.4076 (2)	0.0638 (7)
H22A	0.6643	1.3284	0.3678	0.077*
N3	0.6333 (2)	0.71471 (19)	0.1450 (2)	0.0701 (6)
C6	0.6758 (3)	1.3061 (2)	0.0986 (2)	0.0533 (6)
C5	0.7487 (3)	1.3516 (2)	−0.0066 (2)	0.0653 (7)
H5A	0.6987	1.4194	−0.0391	0.078*
C21	0.7022 (3)	1.2489 (2)	0.5072 (3)	0.0680 (7)
H21A	0.7066	1.3168	0.5329	0.082*
C20	0.7214 (3)	1.1418 (3)	0.5705 (2)	0.0647 (7)
C2	0.8976 (3)	1.1444 (2)	0.0910 (2)	0.0655 (7)
H2A	0.9473	1.0755	0.1228	0.079*
N2	0.7448 (3)	1.1347 (2)	0.6709 (2)	0.0883 (8)
C24	0.6286 (4)	0.6961 (3)	0.0341 (3)	0.0823 (9)
H24A	0.5562	0.7706	0.0218	0.099*
H24B	0.5978	0.6290	0.0417	0.099*
C26	0.6551 (3)	0.6089 (3)	0.2187 (3)	0.0798 (9)
H26A	0.6090	0.5593	0.2096	0.096*
H26B	0.6073	0.6394	0.3015	0.096*
C3	0.9713 (3)	1.1913 (3)	−0.0121 (3)	0.0756 (8)

H3A	1.0718	1.1540	−0.0493	0.091*
C4	0.8970 (3)	1.2929 (3)	−0.0605 (3)	0.0726 (8)
H4A	0.9484	1.3218	−0.1305	0.087*
C8	0.3672 (3)	1.2806 (2)	0.3566 (3)	0.0710 (7)
H8A	0.3801	1.2022	0.3939	0.085*
H8B	0.3024	1.2937	0.3192	0.085*
C30	0.7497 (4)	1.2395 (3)	0.7168 (3)	0.0939 (10)
H30A	0.7281	1.2307	0.8009	0.113*
H30B	0.6741	1.3151	0.7101	0.113*
C27	0.8098 (4)	0.5279 (3)	0.1898 (3)	0.0949 (10)
H27A	0.8156	0.4660	0.2470	0.142*
H27B	0.8585	0.5773	0.1923	0.142*
H27C	0.8551	0.4883	0.1116	0.142*
C28	0.7645 (4)	1.0192 (4)	0.7386 (3)	0.1097 (12)
H28A	0.7429	1.0394	0.8198	0.132*
H28B	0.6983	0.9848	0.7412	0.132*
C31	0.8902 (4)	1.2535 (4)	0.6561 (4)	0.1210 (14)
H31A	0.8822	1.3262	0.6899	0.182*
H31B	0.9135	1.2617	0.5726	0.182*
H31C	0.9651	1.1817	0.6668	0.182*
C25	0.7684 (4)	0.6666 (3)	−0.0731 (3)	0.1066 (12)
H25A	0.7536	0.6651	−0.1433	0.160*
H25B	0.8374	0.5869	−0.0672	0.160*
H25C	0.8044	0.7287	−0.0779	0.160*
C29	0.9134 (5)	0.9280 (5)	0.6809 (4)	0.1498 (18)
H29A	0.9264	0.8558	0.7262	0.225*
H29B	0.9787	0.9632	0.6765	0.225*
H29C	0.9329	0.9051	0.6019	0.225*
C9	0.2953 (4)	1.3869 (4)	0.4548 (3)	0.1138 (13)
H9A	0.3551	1.3705	0.4977	0.137*
H9B	0.2887	1.4649	0.4177	0.137*
C10	0.1492 (5)	1.3971 (5)	0.5393 (4)	0.1599 (19)
H10A	0.1025	1.4693	0.5926	0.240*
H10B	0.1569	1.3245	0.5842	0.240*
H10C	0.0934	1.4041	0.4959	0.240*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.1059 (14)	0.0485 (10)	0.0647 (11)	−0.0381 (9)	−0.0486 (10)	0.0116 (8)
C23	0.0564 (13)	0.0408 (13)	0.0578 (14)	−0.0173 (10)	−0.0287 (11)	0.0015 (10)
C12	0.0576 (14)	0.0424 (13)	0.0595 (14)	−0.0185 (11)	−0.0316 (12)	0.0030 (11)
C17	0.0585 (14)	0.0433 (13)	0.0580 (14)	−0.0183 (11)	−0.0296 (12)	0.0018 (11)
N1	0.0569 (12)	0.0427 (11)	0.0613 (12)	−0.0165 (9)	−0.0274 (10)	0.0039 (9)
C16	0.0707 (16)	0.0426 (13)	0.0670 (16)	−0.0241 (12)	−0.0337 (13)	0.0046 (11)
O1	0.0851 (13)	0.0534 (11)	0.0837 (13)	−0.0134 (10)	−0.0481 (11)	0.0118 (9)
C15	0.0602 (15)	0.0449 (14)	0.0747 (17)	−0.0192 (11)	−0.0350 (13)	−0.0037 (12)
C18	0.0625 (14)	0.0473 (14)	0.0555 (14)	−0.0226 (11)	−0.0279 (12)	0.0016 (11)

C14	0.0780 (17)	0.0527 (15)	0.0790 (18)	−0.0223 (13)	−0.0495 (15)	0.0024 (13)
C11	0.0590 (14)	0.0370 (12)	0.0604 (14)	−0.0165 (10)	−0.0315 (12)	0.0043 (10)
C19	0.0761 (17)	0.0582 (15)	0.0586 (15)	−0.0263 (13)	−0.0357 (13)	0.0066 (12)
C1	0.0636 (15)	0.0440 (13)	0.0592 (14)	−0.0231 (11)	−0.0310 (12)	0.0007 (11)
C7	0.0735 (17)	0.0379 (13)	0.0632 (15)	−0.0183 (12)	−0.0387 (13)	0.0011 (11)
C13	0.0783 (17)	0.0465 (14)	0.0753 (17)	−0.0221 (12)	−0.0493 (15)	0.0105 (12)
C22	0.0762 (17)	0.0500 (15)	0.0777 (18)	−0.0268 (13)	−0.0416 (15)	0.0045 (12)
N3	0.0911 (16)	0.0474 (12)	0.0889 (16)	−0.0300 (11)	−0.0488 (13)	−0.0014 (11)
C6	0.0732 (16)	0.0413 (13)	0.0548 (14)	−0.0243 (12)	−0.0333 (13)	0.0019 (11)
C5	0.094 (2)	0.0524 (15)	0.0645 (17)	−0.0372 (15)	−0.0399 (16)	0.0065 (12)
C21	0.0804 (18)	0.0606 (17)	0.0773 (18)	−0.0289 (14)	−0.0416 (15)	−0.0074 (14)
C20	0.0667 (16)	0.0704 (18)	0.0612 (16)	−0.0243 (13)	−0.0314 (13)	−0.0037 (13)
C2	0.0672 (17)	0.0530 (15)	0.0758 (18)	−0.0201 (13)	−0.0316 (14)	−0.0020 (13)
N2	0.124 (2)	0.0885 (18)	0.0758 (16)	−0.0421 (16)	−0.0630 (16)	0.0043 (14)
C24	0.101 (2)	0.0649 (18)	0.106 (2)	−0.0342 (16)	−0.059 (2)	−0.0102 (16)
C26	0.093 (2)	0.0589 (18)	0.101 (2)	−0.0389 (16)	−0.0416 (18)	−0.0053 (15)
C3	0.0724 (18)	0.083 (2)	0.0725 (19)	−0.0376 (16)	−0.0210 (16)	−0.0096 (16)
C4	0.090 (2)	0.0753 (19)	0.0616 (17)	−0.0474 (17)	−0.0253 (16)	0.0025 (14)
C8	0.0680 (17)	0.0606 (16)	0.0827 (19)	−0.0221 (13)	−0.0356 (15)	0.0125 (14)
C30	0.107 (2)	0.114 (3)	0.078 (2)	−0.047 (2)	−0.0444 (19)	−0.0174 (18)
C27	0.104 (3)	0.071 (2)	0.118 (3)	−0.0303 (18)	−0.056 (2)	−0.0036 (18)
C28	0.107 (3)	0.160 (4)	0.075 (2)	−0.064 (3)	−0.035 (2)	−0.013 (2)
C31	0.121 (3)	0.152 (4)	0.121 (3)	−0.061 (3)	−0.065 (3)	−0.015 (3)
C25	0.144 (3)	0.107 (3)	0.091 (3)	−0.065 (2)	−0.050 (3)	−0.003 (2)
C29	0.104 (3)	0.213 (5)	0.132 (4)	−0.052 (3)	−0.060 (3)	0.005 (3)
C9	0.078 (2)	0.163 (4)	0.070 (2)	−0.037 (2)	−0.0146 (18)	0.003 (2)
C10	0.111 (3)	0.195 (5)	0.124 (4)	−0.031 (3)	−0.033 (3)	0.000 (3)

Geometric parameters (Å, °)

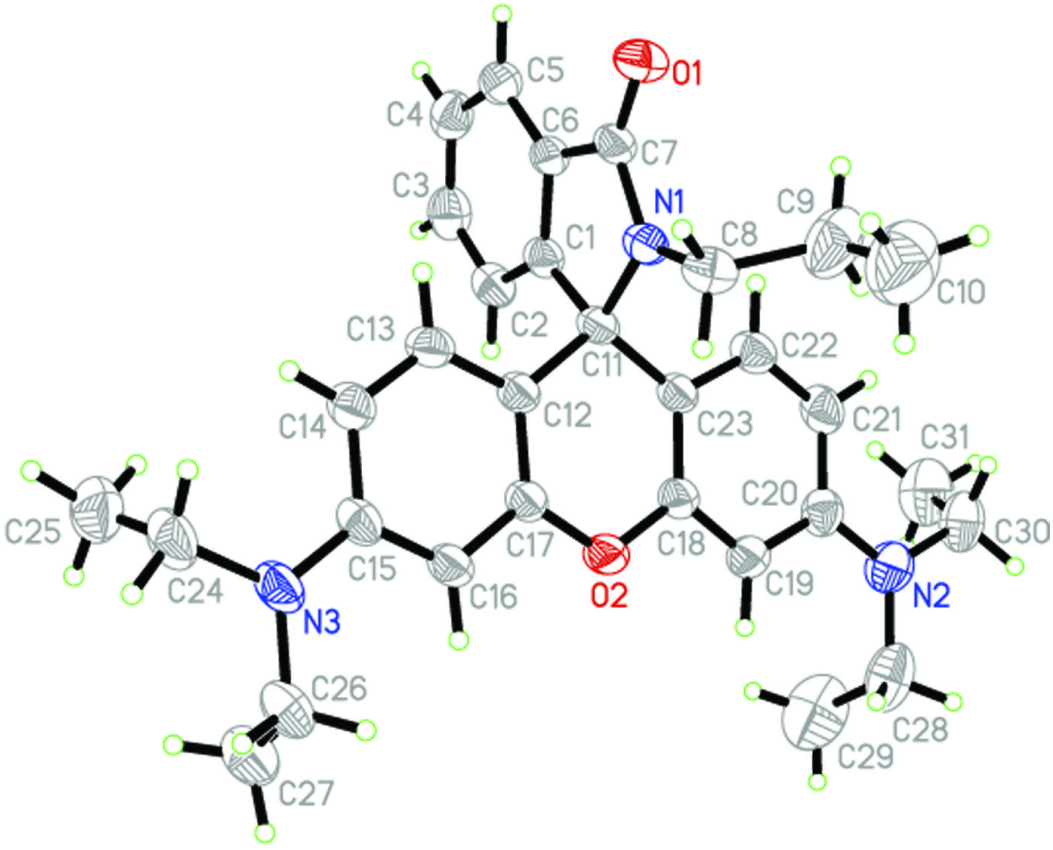
O2—C17	1.377 (3)	N2—C30	1.462 (4)
O2—C18	1.382 (3)	N2—C28	1.503 (5)
C23—C18	1.379 (3)	C24—C25	1.490 (4)
C23—C22	1.396 (3)	C24—H24A	0.9700
C23—C11	1.504 (3)	C24—H24B	0.9700
C12—C17	1.375 (3)	C26—C27	1.490 (4)
C12—C13	1.389 (3)	C26—H26A	0.9700
C12—C11	1.525 (3)	C26—H26B	0.9700
C17—C16	1.388 (3)	C3—C4	1.387 (4)
N1—C7	1.360 (3)	C3—H3A	0.9300
N1—C8	1.434 (3)	C4—H4A	0.9300
N1—C11	1.486 (3)	C8—C9	1.561 (5)
C16—C15	1.388 (3)	C8—H8A	0.9700
C16—H16A	0.9300	C8—H8B	0.9700
O1—C7	1.223 (3)	C30—C31	1.492 (5)
C15—N3	1.382 (3)	C30—H30A	0.9700
C15—C14	1.409 (3)	C30—H30B	0.9700
C18—C19	1.374 (3)	C27—H27A	0.9600
C14—C13	1.364 (3)	C27—H27B	0.9600

C14—H14A	0.9300	C27—H27C	0.9600
C11—C1	1.522 (3)	C28—C29	1.461 (5)
C19—C20	1.402 (3)	C28—H28A	0.9700
C19—H19A	0.9300	C28—H28B	0.9700
C1—C2	1.377 (3)	C31—H31A	0.9600
C1—C6	1.380 (3)	C31—H31B	0.9600
C7—C6	1.472 (3)	C31—H31C	0.9600
C13—H13A	0.9300	C25—H25A	0.9600
C22—C21	1.372 (3)	C25—H25B	0.9600
C22—H22A	0.9300	C25—H25C	0.9600
N3—C24	1.451 (4)	C29—H29A	0.9600
N3—C26	1.459 (4)	C29—H29B	0.9600
C6—C5	1.393 (3)	C29—H29C	0.9600
C5—C4	1.374 (4)	C9—C10	1.476 (5)
C5—H5A	0.9300	C9—H9A	0.9700
C21—C20	1.398 (4)	C9—H9B	0.9700
C21—H21A	0.9300	C10—H10A	0.9600
C20—N2	1.368 (3)	C10—H10B	0.9600
C2—C3	1.389 (4)	C10—H10C	0.9600
C2—H2A	0.9300		
C17—O2—C18	118.45 (18)	N3—C24—H24B	108.6
C18—C23—C22	115.6 (2)	C25—C24—H24B	108.6
C18—C23—C11	123.18 (19)	H24A—C24—H24B	107.5
C22—C23—C11	121.2 (2)	N3—C26—C27	114.2 (3)
C17—C12—C13	116.2 (2)	N3—C26—H26A	108.7
C17—C12—C11	122.7 (2)	C27—C26—H26A	108.7
C13—C12—C11	121.2 (2)	N3—C26—H26B	108.7
C12—C17—O2	123.11 (19)	C27—C26—H26B	108.7
C12—C17—C16	122.1 (2)	H26A—C26—H26B	107.6
O2—C17—C16	114.7 (2)	C2—C3—C4	120.9 (3)
C7—N1—C8	123.7 (2)	C2—C3—H3A	119.5
C7—N1—C11	113.66 (19)	C4—C3—H3A	119.5
C8—N1—C11	122.12 (19)	C5—C4—C3	121.0 (3)
C17—C16—C15	121.4 (2)	C5—C4—H4A	119.5
C17—C16—H16A	119.3	C3—C4—H4A	119.5
C15—C16—H16A	119.3	N1—C8—C9	113.4 (2)
N3—C15—C16	122.1 (2)	N1—C8—H8A	108.9
N3—C15—C14	121.4 (2)	C9—C8—H8A	108.9
C16—C15—C14	116.5 (2)	N1—C8—H8B	108.9
C19—C18—C23	123.3 (2)	C9—C8—H8B	108.9
C19—C18—O2	113.8 (2)	H8A—C8—H8B	107.7
C23—C18—O2	122.9 (2)	N2—C30—C31	115.3 (3)
C13—C14—C15	120.7 (2)	N2—C30—H30A	108.5
C13—C14—H14A	119.6	C31—C30—H30A	108.5
C15—C14—H14A	119.6	N2—C30—H30B	108.5
N1—C11—C23	112.03 (17)	C31—C30—H30B	108.5
N1—C11—C1	100.24 (17)	H30A—C30—H30B	107.5
C23—C11—C1	114.03 (18)	C26—C27—H27A	109.5
N1—C11—C12	110.26 (17)	C26—C27—H27B	109.5

C23—C11—C12	109.73 (18)	H27A—C27—H27B	109.5
C1—C11—C12	110.24 (18)	C26—C27—H27C	109.5
C18—C19—C20	120.5 (2)	H27A—C27—H27C	109.5
C18—C19—H19A	119.7	H27B—C27—H27C	109.5
C20—C19—H19A	119.7	C29—C28—N2	109.8 (3)
C2—C1—C6	120.7 (2)	C29—C28—H28A	109.7
C2—C1—C11	129.0 (2)	N2—C28—H28A	109.7
C6—C1—C11	110.2 (2)	C29—C28—H28B	109.7
O1—C7—N1	125.2 (2)	N2—C28—H28B	109.7
O1—C7—C6	128.1 (2)	H28A—C28—H28B	108.2
N1—C7—C6	106.7 (2)	C30—C31—H31A	109.5
C14—C13—C12	123.1 (2)	C30—C31—H31B	109.5
C14—C13—H13A	118.5	H31A—C31—H31B	109.5
C12—C13—H13A	118.5	C30—C31—H31C	109.5
C21—C22—C23	122.6 (2)	H31A—C31—H31C	109.5
C21—C22—H22A	118.7	H31B—C31—H31C	109.5
C23—C22—H22A	118.7	C24—C25—H25A	109.5
C15—N3—C24	120.9 (2)	C24—C25—H25B	109.5
C15—N3—C26	121.1 (2)	H25A—C25—H25B	109.5
C24—N3—C26	117.3 (2)	C24—C25—H25C	109.5
C1—C6—C5	121.4 (2)	H25A—C25—H25C	109.5
C1—C6—C7	108.8 (2)	H25B—C25—H25C	109.5
C5—C6—C7	129.8 (2)	C28—C29—H29A	109.5
C4—C5—C6	117.8 (3)	C28—C29—H29B	109.5
C4—C5—H5A	121.1	H29A—C29—H29B	109.5
C6—C5—H5A	121.1	C28—C29—H29C	109.5
C22—C21—C20	121.0 (2)	H29A—C29—H29C	109.5
C22—C21—H21A	119.5	H29B—C29—H29C	109.5
C20—C21—H21A	119.5	C10—C9—C8	110.9 (3)
N2—C20—C21	121.6 (2)	C10—C9—H9A	109.5
N2—C20—C19	121.6 (3)	C8—C9—H9A	109.5
C21—C20—C19	116.9 (2)	C10—C9—H9B	109.5
C1—C2—C3	118.2 (3)	C8—C9—H9B	109.5
C1—C2—H2A	120.9	H9A—C9—H9B	108.0
C3—C2—H2A	120.9	C9—C10—H10A	109.5
C20—N2—C30	121.3 (3)	C9—C10—H10B	109.5
C20—N2—C28	121.1 (2)	H10A—C10—H10B	109.5
C30—N2—C28	117.6 (3)	C9—C10—H10C	109.5
N3—C24—C25	114.8 (2)	H10A—C10—H10C	109.5
N3—C24—H24A	108.6	H10B—C10—H10C	109.5
C25—C24—H24A	108.6		
C13—C12—C17—O2	179.4 (2)	C8—N1—C7—C6	−175.5 (2)
C11—C12—C17—O2	−0.8 (4)	C11—N1—C7—C6	−3.4 (2)
C13—C12—C17—C16	0.4 (3)	C15—C14—C13—C12	0.9 (4)
C11—C12—C17—C16	−179.8 (2)	C17—C12—C13—C14	−0.5 (4)
C18—O2—C17—C12	−0.2 (3)	C11—C12—C13—C14	179.7 (2)
C18—O2—C17—C16	178.9 (2)	C18—C23—C22—C21	0.5 (4)
C12—C17—C16—C15	−0.7 (4)	C11—C23—C22—C21	−179.7 (2)
O2—C17—C16—C15	−179.8 (2)	C16—C15—N3—C24	−172.4 (2)

C17—C16—C15—N3	−178.0 (2)	C14—C15—N3—C24	8.6 (4)
C17—C16—C15—C14	1.1 (4)	C16—C15—N3—C26	−2.0 (4)
C22—C23—C18—C19	−1.1 (4)	C14—C15—N3—C26	178.9 (2)
C11—C23—C18—C19	179.1 (2)	C2—C1—C6—C5	2.7 (3)
C22—C23—C18—O2	179.9 (2)	C11—C1—C6—C5	−175.1 (2)
C11—C23—C18—O2	0.0 (4)	C2—C1—C6—C7	−177.88 (19)
C17—O2—C18—C19	−178.6 (2)	C11—C1—C6—C7	4.3 (2)
C17—O2—C18—C23	0.5 (3)	O1—C7—C6—C1	179.2 (2)
N3—C15—C14—C13	178.0 (2)	N1—C7—C6—C1	−0.6 (2)
C16—C15—C14—C13	−1.1 (4)	O1—C7—C6—C5	−1.5 (4)
C7—N1—C11—C23	126.9 (2)	N1—C7—C6—C5	178.7 (2)
C8—N1—C11—C23	−60.9 (3)	C1—C6—C5—C4	−2.4 (3)
C7—N1—C11—C1	5.6 (2)	C7—C6—C5—C4	178.3 (2)
C8—N1—C11—C1	177.8 (2)	C23—C22—C21—C20	−0.2 (4)
C7—N1—C11—C12	−110.6 (2)	C22—C21—C20—N2	−179.2 (2)
C8—N1—C11—C12	61.6 (3)	C22—C21—C20—C19	0.5 (4)
C18—C23—C11—N1	122.0 (2)	C18—C19—C20—N2	178.6 (2)
C22—C23—C11—N1	−57.9 (3)	C18—C19—C20—C21	−1.1 (4)
C18—C23—C11—C1	−125.1 (2)	C6—C1—C2—C3	−1.0 (3)
C22—C23—C11—C1	55.1 (3)	C11—C1—C2—C3	176.5 (2)
C18—C23—C11—C12	−0.8 (3)	C21—C20—N2—C30	0.5 (4)
C22—C23—C11—C12	179.3 (2)	C19—C20—N2—C30	−179.2 (3)
C17—C12—C11—N1	−122.7 (2)	C21—C20—N2—C28	179.7 (3)
C13—C12—C11—N1	57.2 (3)	C19—C20—N2—C28	0.0 (4)
C17—C12—C11—C23	1.2 (3)	C15—N3—C24—C25	74.5 (3)
C13—C12—C11—C23	−179.0 (2)	C26—N3—C24—C25	−96.2 (3)
C17—C12—C11—C1	127.6 (2)	C15—N3—C26—C27	−82.3 (3)
C13—C12—C11—C1	−52.6 (3)	C24—N3—C26—C27	88.4 (3)
C23—C18—C19—C20	1.5 (4)	C1—C2—C3—C4	−1.0 (4)
O2—C18—C19—C20	−179.4 (2)	C6—C5—C4—C3	0.4 (4)
N1—C11—C1—C2	176.6 (2)	C2—C3—C4—C5	1.3 (4)
C23—C11—C1—C2	56.7 (3)	C7—N1—C8—C9	−77.3 (3)
C12—C11—C1—C2	−67.2 (3)	C11—N1—C8—C9	111.3 (3)
N1—C11—C1—C6	−5.8 (2)	C20—N2—C30—C31	−81.0 (4)
C23—C11—C1—C6	−125.7 (2)	C28—N2—C30—C31	99.8 (4)
C12—C11—C1—C6	110.4 (2)	C20—N2—C28—C29	81.2 (4)
C8—N1—C7—O1	4.7 (4)	C30—N2—C28—C29	−99.6 (3)
C11—N1—C7—O1	176.7 (2)	N1—C8—C9—C10	175.2 (3)

Fig. 1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