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# 3′,6′-Bis(diethylamino)-2-[(*E*)-2-(4-hydroxy-3-methoxybenzylideneamino)-ethyl]spiro[isoindoline-1,9′-xanthen]-3-one ethanol monosolvate

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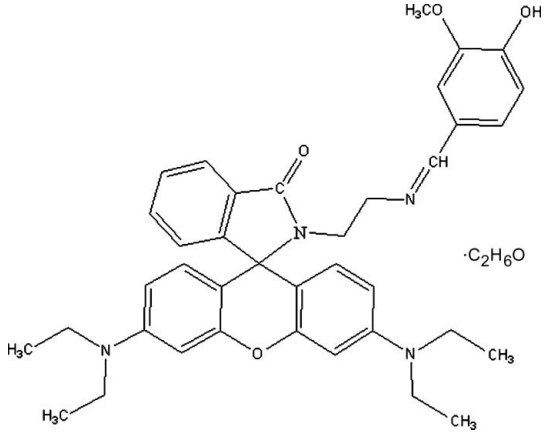
Key indicators: single-crystal X-ray study; $T$ = 113 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.049; $wR$ factor = 0.119; data-to-parameter ratio = 19.1.

In the title compound, $C_{38}H_{42}N_4O_4\cdot C_2H_6O$, prepared *via* a spirolactam ring-formation reaction in a rhodamine dye, the xanthene ring system is approximately planar (r.m.s. deviation = 0.0014Å) and subtends dihedral angles of 88.10 (3) and 86.92 (4)° with the spirolactam (r.m.s. deviations = 0.0012 Å) and benzene rings, respectively. The crystal structure consists of chains parallel to [$\bar{1}$01], formed *via* O—H···O interactions.

## Related literature

For related structures and background to rhodamine-based dyes, see: Xu *et al.*(2010*a*,*b*); Zhang *et al.* (2008); Tian & Peng (2008); Kwon *et al.* (2005); Wu *et al.*(2007).




## Experimental

### Crystal data

$C_{38}H_{42}N_4O_4\cdot C_2H_6O$
$M_r$ = 664.82
Monoclinic, $P2_1/n$
$a$ = 16.674 (4) Å
$b$ = 12.197 (3) Å
$c$ = 17.936 (4) Å
$\beta$ = 96.445 (4)°
$V$ = 3624.5 (15) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.08 mm$^{-1}$
$T$ = 113 K
0.20 × 0.12 × 0.10 mm

### Data collection

Rigaku Saturn724+ diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2008)
$T_{min}$ = 0.984, $T_{max}$ = 0.992
45486 measured reflections
8576 independent reflections
7385 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.043

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.119
$S$ = 1.10
8576 reflections
450 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.21 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O3—H3···O5$^{i}$ | 0.84 | 1.96 | 2.7052 (15) | 148 |
| O5—H5$A$···O2 | 0.84 | 1.93 | 2.7552 (14) | 166 |

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

Data collection: *CrystalClear* (Rigaku, 2008); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *CrystalStructure* (Rigaku, 2008).

The authors thank the Science Development Committee of Tianjin Agricultural University (research grant No. 2009 N02).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2444).

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# supporting information

*Acta Cryst.* (2012). E**68**, o1479 [doi:10.1107/S1600536812016741]

## 3′,6′-Bis(diethylamino)-2-[(*E*)-2-(4-hydroxy-3-methoxybenzylideneamino)ethyl]-spiro[isoindoline-1,9′-xanthen]-3-one ethanol monosolvate

**Zhen Wei, Jinlong Guo, Xujun Zheng, Shunwei Chen and Qun Wan**

### S1. Comment

Rhodamine-based dyes have been widely used for conjugation with biomolecules, owing to their excellent spectroscopic properties such as large molar extinction coefficient and high fluorescence quantum yields. Moreover, it is well known that many derivatives of Rhodamine undergo equilibrium between spirolactam and an open ring amide, and both conformations behave with different spectroscopic properties, for what Rhodamine-based dyes have been widely used as sensing materials (Kwon *et al.*, 2005). Detailed information on their molecular and crystal structures is necessary to understand their photophysical and photochemical properties. In the title compound [($C_{38}H_{42}N_4O_4$)], the xanthene and the spirolactam rings are almost planar (r.m.s. deviations from the mean plane, 0.0014Å and 0.0012 Å, respectively), with the former ring forming dihedral angles of 88.10 (3)° to the spirolactam ring and 86.92 (4)°. to the benzene ring. The crystal structure consists of one-dimensional chains parallel to [$\bar{1}$01], formed *via* O3—H3···O5, O5—H5A···O2 interactions (Table 1).

### S2. Experimental

*N*-(rhodamine-6 G)lactam-ethylenediamine (5*m* mol) was dissolved in 50 ml of ethanol, followed by addition of 3-methoxy-4-hydroxybenzaldehyde(5*m* mol). The solution was stirred and refluxed for 10 h. The white precipitate was filtrated and disolved in ethanol. Single crystals suitable for X-ray measurements were obtained at room temperature by slow evaporation of this solution.

### S3. Refinement

All H atoms were seen in the final diffreence map, but further replaced at their expected positions and treated as riding on their parent atoms, with C—H = 0.93 Å for the aromatic, 0.96 Å for the methyl and C—H= 0.97 Å for methylene H atoms, and O—H: 0.84Å. In al cases $U_{iso}$(H)= x×$U_{eq}$(Host) with x = 1.2 except for methyl groups in which x = 1.5.

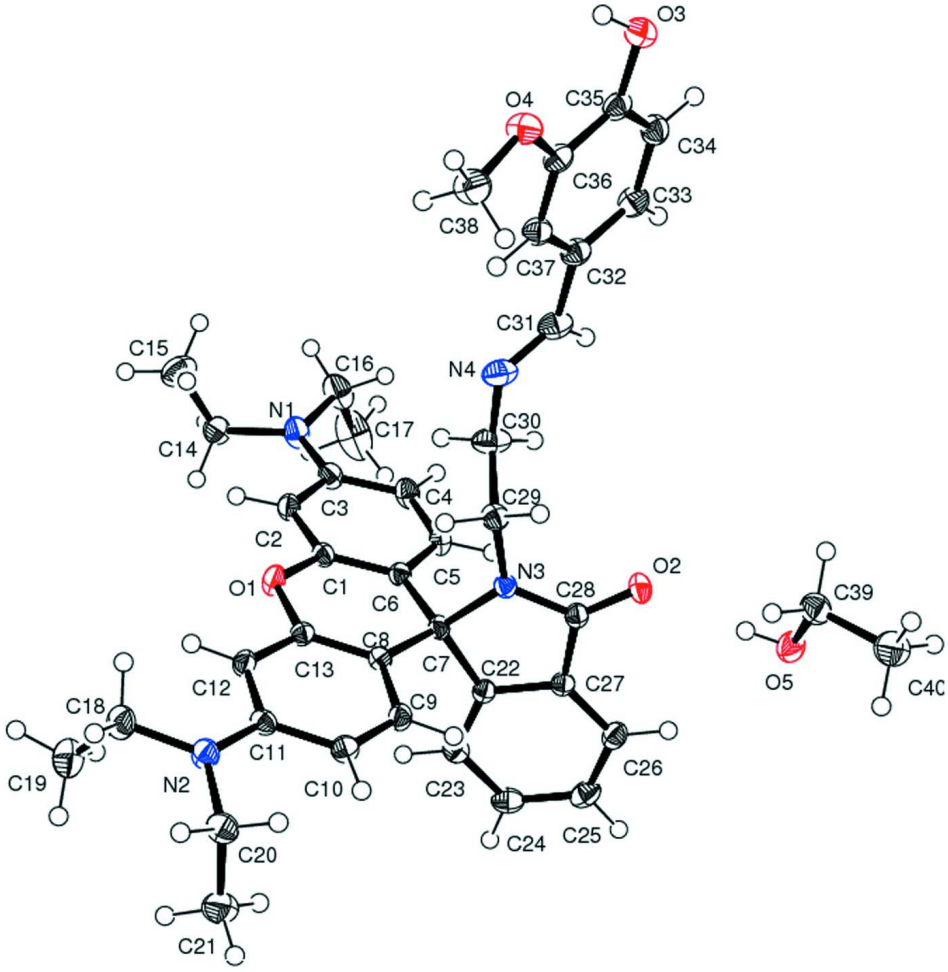


**Figure 1**

The molecular structure of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

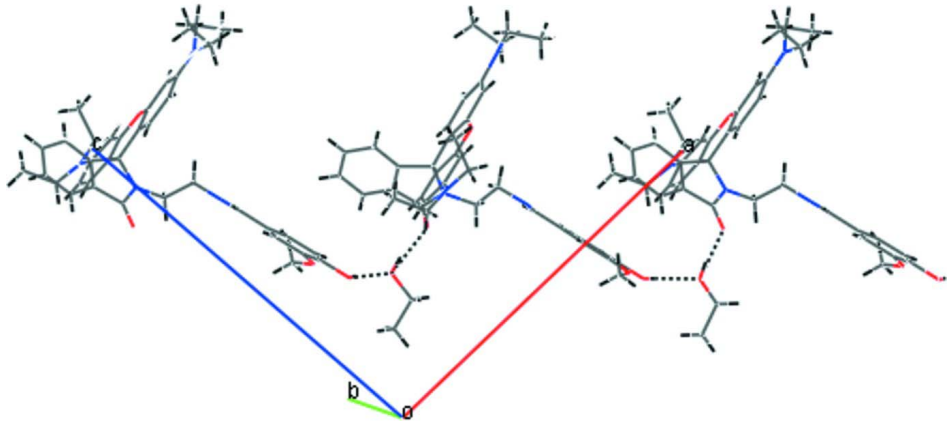


**Figure 2**

The one dimentional structure formed by the inter-molecular hydrogen bonds drawn in dashed lines

**3′,6′-Bis(diethylamino)-2-[(*E*)-2-(4-hydroxy-3- methoxybenzylideneamino)ethyl]spiro[isoindoline-1,9′-xanthen]-3-one ethanol monosolvate**

*Crystal data*

$C_{38}H_{42}N_4O_4 \cdot C_2H_6O$
$M_r = 664.82$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 16.674$ (4) Å
$b = 12.197$ (3) Å
$c = 17.936$ (4) Å
$\beta = 96.445$ (4)°
$V = 3624.5$ (15) Å$^3$
$Z = 4$
$F(000) = 1424$
$D_x = 1.218$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 10662 reflections
$\theta = 1.7$–27.9°
$\mu = 0.08$ mm$^{-1}$
$T = 113$ K
Prism, colourless
0.20 × 0.12 × 0.10 mm

*Data collection*

Rigaku Saturn724+ diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 28.5714 pixels mm$^{-1}$
profile data from $\omega$–scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2008)
$T_{min} = 0.984$, $T_{max} = 0.992$
45486 measured reflections
8576 independent reflections
7385 reflections with $I > 2\sigma(I)$
$R_{int} = 0.043$
$\theta_{max} = 27.8°$, $\theta_{min} = 1.6°$
$h = -21 \rightarrow 20$
$k = -16 \rightarrow 15$
$l = -23 \rightarrow 22$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.119$
$S = 1.10$
8576 reflections
450 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0532P)^2 + 0.5173P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.32$ e Å$^{-3}$
$\Delta\rho_{min} = -0.21$ e Å$^{-3}$

*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**. 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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.70816 (5) | 0.41422 (7) | 0.34461 (6) | 0.0243 (2) |
| O2 | 0.45673 (6) | 0.76065 (8) | 0.19829 (5) | 0.0257 (2) |
| O3 | 0.72460 (6) | 0.76249 (8) | −0.25561 (5) | 0.0281 (2) |
| H3 | 0.7427 | 0.7031 | −0.2705 | 0.042* |
| O4 | 0.70162 (6) | 0.56284 (8) | −0.19741 (5) | 0.0284 (2) |
| N1 | 0.91108 (7) | 0.68357 (10) | 0.39924 (7) | 0.0279 (3) |
| N2 | 0.53054 (7) | 0.10821 (9) | 0.31672 (7) | 0.0238 (3) |
| N3 | 0.54525 (6) | 0.62774 (9) | 0.24766 (6) | 0.0171 (2) |
| N4 | 0.66575 (7) | 0.64858 (11) | 0.08517 (7) | 0.0296 (3) |
| C1 | 0.72358 (8) | 0.52500 (10) | 0.35283 (7) | 0.0185 (3) |
| C2 | 0.80483 (8) | 0.55002 (11) | 0.37132 (7) | 0.0210 (3) |
| H2 | 0.8432 | 0.4923 | 0.3787 | 0.025* |
| C3 | 0.83095 (8) | 0.65951 (11) | 0.37922 (7) | 0.0217 (3) |
| C4 | 0.77103 (8) | 0.74200 (11) | 0.36664 (8) | 0.0239 (3) |
| H4A | 0.7862 | 0.8171 | 0.3700 | 0.029* |
| C5 | 0.69080 (8) | 0.71404 (11) | 0.34955 (7) | 0.0215 (3) |
| H5 | 0.6519 | 0.7711 | 0.3423 | 0.026* |
| C6 | 0.66416 (7) | 0.60495 (10) | 0.34241 (7) | 0.0171 (3) |
| C7 | 0.57572 (7) | 0.57688 (10) | 0.32152 (7) | 0.0168 (3) |
| C8 | 0.56436 (7) | 0.45356 (10) | 0.31992 (7) | 0.0172 (3) |
| C9 | 0.48720 (8) | 0.40554 (11) | 0.30829 (7) | 0.0197 (3) |
| H9 | 0.4414 | 0.4522 | 0.3007 | 0.024* |
| C10 | 0.47538 (8) | 0.29366 (11) | 0.30742 (7) | 0.0209 (3) |
| H10 | 0.4220 | 0.2653 | 0.2997 | 0.025* |
| C11 | 0.54168 (8) | 0.22038 (10) | 0.31793 (7) | 0.0193 (3) |
| C12 | 0.61892 (8) | 0.26706 (10) | 0.32955 (7) | 0.0200 (3) |
| H12 | 0.6650 | 0.2209 | 0.3368 | 0.024* |
| C13 | 0.62862 (7) | 0.38069 (10) | 0.33055 (7) | 0.0181 (3) |
| C14 | 0.96754 (8) | 0.59920 (12) | 0.42960 (8) | 0.0268 (3) |
| H14A | 0.9384 | 0.5463 | 0.4586 | 0.032* |
| H14B | 1.0101 | 0.6340 | 0.4648 | 0.032* |
| C15 | 1.00743 (9) | 0.53699 (15) | 0.37037 (9) | 0.0380 (4) |

| | | | | |
|---|---|---|---|---|
| H15A | 0.9662 | 0.4977 | 0.3374 | 0.057* |
| H15B | 1.0462 | 0.4843 | 0.3948 | 0.057* |
| H15C | 1.0356 | 0.5888 | 0.3406 | 0.057* |
| C16 | 0.94234 (9) | 0.79470 (13) | 0.39605 (9) | 0.0361 (4) |
| H16A | 0.9114 | 0.8341 | 0.3540 | 0.043* |
| H16B | 0.9993 | 0.7913 | 0.3855 | 0.043* |
| C17 | 0.93788 (13) | 0.85921 (15) | 0.46778 (11) | 0.0575 (6) |
| H17A | 0.8812 | 0.8693 | 0.4760 | 0.086* |
| H17B | 0.9634 | 0.9310 | 0.4635 | 0.086* |
| H17C | 0.9661 | 0.8190 | 0.5102 | 0.086* |
| C18 | 0.59972 (9) | 0.03336 (11) | 0.32402 (8) | 0.0277 (3) |
| H18A | 0.6422 | 0.0644 | 0.2959 | 0.033* |
| H18B | 0.5826 | −0.0373 | 0.3002 | 0.033* |
| C19 | 0.63582 (10) | 0.01127 (14) | 0.40393 (9) | 0.0370 (4) |
| H19A | 0.6489 | 0.0810 | 0.4296 | 0.055* |
| H19B | 0.6851 | −0.0324 | 0.4035 | 0.055* |
| H19C | 0.5968 | −0.0291 | 0.4304 | 0.055* |
| C20 | 0.45004 (9) | 0.06076 (11) | 0.31775 (8) | 0.0270 (3) |
| H20A | 0.4513 | −0.0164 | 0.3007 | 0.032* |
| H20B | 0.4117 | 0.1010 | 0.2815 | 0.032* |
| C21 | 0.41881 (10) | 0.06364 (13) | 0.39453 (9) | 0.0366 (4) |
| H21A | 0.4540 | 0.0192 | 0.4300 | 0.055* |
| H21B | 0.3638 | 0.0340 | 0.3904 | 0.055* |
| H21C | 0.4185 | 0.1395 | 0.4125 | 0.055* |
| C22 | 0.52088 (7) | 0.63452 (10) | 0.37207 (7) | 0.0169 (3) |
| C23 | 0.51696 (8) | 0.62006 (11) | 0.44828 (7) | 0.0216 (3) |
| H23 | 0.5518 | 0.5701 | 0.4767 | 0.026* |
| C24 | 0.46017 (9) | 0.68132 (12) | 0.48185 (8) | 0.0261 (3) |
| H24 | 0.4563 | 0.6729 | 0.5340 | 0.031* |
| C25 | 0.40883 (8) | 0.75484 (12) | 0.44026 (8) | 0.0263 (3) |
| H25 | 0.3702 | 0.7950 | 0.4643 | 0.032* |
| C26 | 0.41351 (8) | 0.76991 (11) | 0.36424 (8) | 0.0228 (3) |
| H26 | 0.3791 | 0.8204 | 0.3358 | 0.027* |
| C27 | 0.47024 (7) | 0.70856 (10) | 0.33102 (7) | 0.0181 (3) |
| C28 | 0.48748 (7) | 0.70542 (10) | 0.25194 (7) | 0.0188 (3) |
| C29 | 0.57599 (8) | 0.59905 (11) | 0.17751 (7) | 0.0216 (3) |
| H29A | 0.5332 | 0.6123 | 0.1356 | 0.026* |
| H29B | 0.5889 | 0.5198 | 0.1781 | 0.026* |
| C30 | 0.65106 (9) | 0.66352 (14) | 0.16326 (8) | 0.0326 (4) |
| H30A | 0.6431 | 0.7423 | 0.1735 | 0.039* |
| H30B | 0.6982 | 0.6372 | 0.1971 | 0.039* |
| C31 | 0.67072 (8) | 0.73611 (13) | 0.04736 (8) | 0.0269 (3) |
| H31 | 0.6655 | 0.8039 | 0.0724 | 0.032* |
| C32 | 0.68390 (8) | 0.73984 (12) | −0.03207 (8) | 0.0237 (3) |
| C33 | 0.69533 (9) | 0.84087 (12) | −0.06541 (8) | 0.0266 (3) |
| H33 | 0.6938 | 0.9060 | −0.0366 | 0.032* |
| C34 | 0.70888 (8) | 0.84776 (12) | −0.14018 (8) | 0.0255 (3) |
| H34 | 0.7161 | 0.9172 | −0.1624 | 0.031* |

| | | | | |
|---|---|---|---|---|
| C35 | 0.71177 (8) | 0.75289 (12) | −0.18223 (8) | 0.0228 (3) |
| C36 | 0.69883 (8) | 0.65032 (11) | −0.14962 (8) | 0.0222 (3) |
| C37 | 0.68563 (8) | 0.64348 (12) | −0.07481 (8) | 0.0234 (3) |
| H37 | 0.6778 | 0.5741 | −0.0526 | 0.028* |
| C38 | 0.67573 (10) | 0.45925 (12) | −0.17106 (9) | 0.0336 (4) |
| H38A | 0.7124 | 0.4361 | −0.1274 | 0.050* |
| H38B | 0.6761 | 0.4045 | −0.2110 | 0.050* |
| H38C | 0.6209 | 0.4663 | −0.1568 | 0.050* |
| O5 | 0.31286 (6) | 0.87055 (9) | 0.16462 (5) | 0.0278 (2) |
| H5A | 0.3581 | 0.8447 | 0.1812 | 0.042* |
| C39 | 0.29463 (9) | 0.84136 (13) | 0.08727 (8) | 0.0290 (3) |
| H39A | 0.3038 | 0.7619 | 0.0808 | 0.035* |
| H39B | 0.3306 | 0.8819 | 0.0566 | 0.035* |
| C40 | 0.20807 (9) | 0.86893 (15) | 0.06150 (9) | 0.0371 (4) |
| H40A | 0.1727 | 0.8273 | 0.0912 | 0.056* |
| H40B | 0.1959 | 0.8499 | 0.0084 | 0.056* |
| H40C | 0.1993 | 0.9476 | 0.0682 | 0.056* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0147 (5) | 0.0144 (5) | 0.0434 (6) | −0.0003 (4) | 0.0010 (4) | 0.0004 (4) |
| O2 | 0.0234 (5) | 0.0265 (5) | 0.0263 (5) | 0.0051 (4) | −0.0012 (4) | 0.0095 (4) |
| O3 | 0.0307 (6) | 0.0294 (6) | 0.0247 (5) | 0.0006 (4) | 0.0055 (4) | 0.0024 (4) |
| O4 | 0.0348 (6) | 0.0240 (5) | 0.0277 (5) | −0.0042 (4) | 0.0088 (4) | −0.0042 (4) |
| N1 | 0.0187 (6) | 0.0229 (6) | 0.0401 (7) | −0.0045 (5) | −0.0060 (5) | 0.0068 (5) |
| N2 | 0.0228 (6) | 0.0165 (6) | 0.0320 (7) | −0.0016 (5) | 0.0029 (5) | −0.0009 (5) |
| N3 | 0.0175 (5) | 0.0176 (5) | 0.0158 (5) | 0.0012 (4) | 0.0009 (4) | 0.0010 (4) |
| N4 | 0.0242 (6) | 0.0417 (8) | 0.0238 (6) | −0.0021 (5) | 0.0067 (5) | −0.0018 (5) |
| C1 | 0.0188 (6) | 0.0152 (6) | 0.0214 (7) | −0.0003 (5) | 0.0021 (5) | 0.0024 (5) |
| C2 | 0.0175 (6) | 0.0188 (6) | 0.0263 (7) | 0.0017 (5) | 0.0005 (5) | 0.0029 (5) |
| C3 | 0.0192 (7) | 0.0223 (7) | 0.0226 (7) | −0.0023 (5) | −0.0018 (5) | 0.0033 (5) |
| C4 | 0.0241 (7) | 0.0163 (6) | 0.0297 (8) | −0.0033 (5) | −0.0039 (6) | 0.0017 (5) |
| C5 | 0.0212 (7) | 0.0174 (6) | 0.0249 (7) | 0.0023 (5) | −0.0019 (5) | 0.0010 (5) |
| C6 | 0.0165 (6) | 0.0177 (6) | 0.0168 (6) | 0.0002 (5) | 0.0008 (5) | 0.0010 (5) |
| C7 | 0.0162 (6) | 0.0176 (6) | 0.0162 (6) | 0.0015 (5) | −0.0005 (5) | 0.0023 (5) |
| C8 | 0.0182 (6) | 0.0173 (6) | 0.0160 (6) | 0.0001 (5) | 0.0016 (5) | 0.0009 (5) |
| C9 | 0.0171 (6) | 0.0202 (6) | 0.0213 (7) | 0.0019 (5) | −0.0004 (5) | 0.0017 (5) |
| C10 | 0.0174 (6) | 0.0211 (7) | 0.0235 (7) | −0.0029 (5) | −0.0003 (5) | 0.0000 (5) |
| C11 | 0.0236 (7) | 0.0160 (6) | 0.0186 (7) | −0.0016 (5) | 0.0033 (5) | −0.0001 (5) |
| C12 | 0.0173 (6) | 0.0167 (6) | 0.0264 (7) | 0.0025 (5) | 0.0037 (5) | 0.0004 (5) |
| C13 | 0.0151 (6) | 0.0187 (6) | 0.0205 (7) | −0.0008 (5) | 0.0019 (5) | 0.0004 (5) |
| C14 | 0.0183 (7) | 0.0326 (8) | 0.0282 (8) | −0.0019 (6) | −0.0029 (6) | 0.0057 (6) |
| C15 | 0.0228 (8) | 0.0558 (11) | 0.0354 (9) | 0.0019 (7) | 0.0030 (7) | 0.0001 (8) |
| C16 | 0.0276 (8) | 0.0291 (8) | 0.0482 (10) | −0.0131 (6) | −0.0102 (7) | 0.0110 (7) |
| C17 | 0.0718 (14) | 0.0336 (10) | 0.0585 (12) | −0.0136 (9) | −0.0296 (10) | −0.0004 (9) |
| C18 | 0.0293 (8) | 0.0163 (7) | 0.0375 (8) | 0.0002 (6) | 0.0038 (6) | −0.0029 (6) |
| C19 | 0.0392 (9) | 0.0311 (8) | 0.0402 (9) | 0.0072 (7) | 0.0026 (7) | −0.0003 (7) |

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|---|---|---|---|---|---|---|
| C20 | 0.0267 (7) | 0.0185 (7) | 0.0357 (8) | −0.0064 (6) | 0.0037 (6) | −0.0004 (6) |
| C21 | 0.0364 (9) | 0.0316 (9) | 0.0440 (10) | −0.0022 (7) | 0.0142 (7) | 0.0034 (7) |
| C22 | 0.0150 (6) | 0.0153 (6) | 0.0202 (7) | −0.0015 (5) | 0.0009 (5) | −0.0009 (5) |
| C23 | 0.0220 (7) | 0.0223 (7) | 0.0201 (7) | −0.0021 (5) | 0.0005 (5) | 0.0007 (5) |
| C24 | 0.0282 (8) | 0.0307 (8) | 0.0202 (7) | −0.0042 (6) | 0.0068 (6) | −0.0033 (6) |
| C25 | 0.0225 (7) | 0.0252 (7) | 0.0324 (8) | 0.0001 (6) | 0.0084 (6) | −0.0069 (6) |
| C26 | 0.0172 (6) | 0.0189 (7) | 0.0323 (8) | 0.0001 (5) | 0.0026 (6) | −0.0005 (5) |
| C27 | 0.0153 (6) | 0.0168 (6) | 0.0220 (7) | −0.0020 (5) | 0.0014 (5) | 0.0004 (5) |
| C28 | 0.0143 (6) | 0.0169 (6) | 0.0244 (7) | −0.0020 (5) | −0.0004 (5) | 0.0021 (5) |
| C29 | 0.0249 (7) | 0.0219 (7) | 0.0181 (7) | 0.0006 (5) | 0.0029 (5) | −0.0012 (5) |
| C30 | 0.0277 (8) | 0.0497 (10) | 0.0211 (7) | −0.0089 (7) | 0.0060 (6) | −0.0027 (7) |
| C31 | 0.0218 (7) | 0.0338 (8) | 0.0254 (8) | −0.0055 (6) | 0.0041 (6) | −0.0063 (6) |
| C32 | 0.0187 (7) | 0.0281 (7) | 0.0243 (7) | −0.0015 (5) | 0.0025 (5) | −0.0015 (6) |
| C33 | 0.0268 (7) | 0.0245 (7) | 0.0290 (8) | 0.0002 (6) | 0.0049 (6) | −0.0054 (6) |
| C34 | 0.0242 (7) | 0.0219 (7) | 0.0304 (8) | 0.0005 (6) | 0.0031 (6) | 0.0023 (6) |
| C35 | 0.0159 (6) | 0.0293 (7) | 0.0230 (7) | 0.0008 (5) | 0.0013 (5) | −0.0013 (6) |
| C36 | 0.0177 (6) | 0.0246 (7) | 0.0243 (7) | −0.0006 (5) | 0.0023 (5) | −0.0034 (5) |
| C37 | 0.0201 (7) | 0.0235 (7) | 0.0269 (8) | −0.0023 (5) | 0.0037 (6) | 0.0010 (5) |
| C38 | 0.0377 (9) | 0.0229 (7) | 0.0413 (9) | −0.0045 (6) | 0.0099 (7) | −0.0035 (6) |
| O5 | 0.0232 (5) | 0.0368 (6) | 0.0229 (5) | 0.0051 (4) | −0.0004 (4) | 0.0013 (4) |
| C39 | 0.0315 (8) | 0.0323 (8) | 0.0229 (7) | 0.0023 (6) | 0.0019 (6) | 0.0018 (6) |
| C40 | 0.0296 (8) | 0.0501 (10) | 0.0300 (8) | −0.0048 (7) | −0.0039 (7) | 0.0052 (7) |

*Geometric parameters (Å, º)*

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| O1—C1 | 1.3802 (15) | C18—C19 | 1.515 (2) |
| O1—C13 | 1.3836 (15) | C18—H18A | 0.9900 |
| O2—C28 | 1.2376 (15) | C18—H18B | 0.9900 |
| O3—C35 | 1.3619 (17) | C19—H19A | 0.9800 |
| O3—H3 | 0.8400 | C19—H19B | 0.9800 |
| O4—C36 | 1.3726 (16) | C19—H19C | 0.9800 |
| O4—C38 | 1.4324 (17) | C20—C21 | 1.527 (2) |
| N1—C3 | 1.3756 (17) | C20—H20A | 0.9900 |
| N1—C16 | 1.4556 (18) | C20—H20B | 0.9900 |
| N1—C14 | 1.4583 (18) | C21—H21A | 0.9800 |
| N2—C11 | 1.3805 (17) | C21—H21B | 0.9800 |
| N2—C20 | 1.4636 (18) | C21—H21C | 0.9800 |
| N2—C18 | 1.4653 (18) | C22—C23 | 1.3871 (18) |
| N3—C28 | 1.3593 (16) | C22—C27 | 1.3895 (18) |
| N3—C29 | 1.4537 (16) | C23—C24 | 1.3949 (19) |
| N3—C7 | 1.4989 (16) | C23—H23 | 0.9500 |
| N4—C31 | 1.2724 (19) | C24—C25 | 1.396 (2) |
| N4—C30 | 1.4603 (18) | C24—H24 | 0.9500 |
| C1—C6 | 1.3877 (18) | C25—C26 | 1.387 (2) |
| C1—C2 | 1.3919 (18) | C25—H25 | 0.9500 |
| C2—C3 | 1.4068 (18) | C26—C27 | 1.3916 (18) |
| C2—H2 | 0.9500 | C26—H26 | 0.9500 |
| C3—C4 | 1.4180 (19) | C27—C28 | 1.4790 (18) |

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| C4—C5 | 1.3812 (19) | C29—C30 | 1.524 (2) |
| C4—H4A | 0.9500 | C29—H29A | 0.9900 |
| C5—C6 | 1.4041 (18) | C29—H29B | 0.9900 |
| C5—H5 | 0.9500 | C30—H30A | 0.9900 |
| C6—C7 | 1.5193 (17) | C30—H30B | 0.9900 |
| C7—C8 | 1.5159 (18) | C31—C32 | 1.466 (2) |
| C7—C22 | 1.5292 (17) | C31—H31 | 0.9500 |
| C8—C13 | 1.3887 (18) | C32—C33 | 1.392 (2) |
| C8—C9 | 1.4077 (18) | C32—C37 | 1.4052 (19) |
| C9—C10 | 1.3786 (18) | C33—C34 | 1.387 (2) |
| C9—H9 | 0.9500 | C33—H33 | 0.9500 |
| C10—C11 | 1.4176 (19) | C34—C35 | 1.3851 (19) |
| C10—H10 | 0.9500 | C34—H34 | 0.9500 |
| C11—C12 | 1.4021 (18) | C35—C36 | 1.4078 (19) |
| C12—C13 | 1.3952 (18) | C36—C37 | 1.3865 (19) |
| C12—H12 | 0.9500 | C37—H37 | 0.9500 |
| C14—C15 | 1.518 (2) | C38—H38A | 0.9800 |
| C14—H14A | 0.9900 | C38—H38B | 0.9800 |
| C14—H14B | 0.9900 | C38—H38C | 0.9800 |
| C15—H15A | 0.9800 | O5—C39 | 1.4317 (17) |
| C15—H15B | 0.9800 | O5—H5A | 0.8400 |
| C15—H15C | 0.9800 | C39—C40 | 1.503 (2) |
| C16—C17 | 1.517 (3) | C39—H39A | 0.9900 |
| C16—H16A | 0.9900 | C39—H39B | 0.9900 |
| C16—H16B | 0.9900 | C40—H40A | 0.9800 |
| C17—H17A | 0.9800 | C40—H40B | 0.9800 |
| C17—H17B | 0.9800 | C40—H40C | 0.9800 |
| C17—H17C | 0.9800 | | |

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| C1—O1—C13 | 118.17 (10) | C18—C19—H19C | 109.5 |
| C35—O3—H3 | 109.5 | H19A—C19—H19C | 109.5 |
| C36—O4—C38 | 116.56 (11) | H19B—C19—H19C | 109.5 |
| C3—N1—C16 | 122.01 (12) | N2—C20—C21 | 114.11 (12) |
| C3—N1—C14 | 120.95 (12) | N2—C20—H20A | 108.7 |
| C16—N1—C14 | 116.92 (11) | C21—C20—H20A | 108.7 |
| C11—N2—C20 | 120.91 (11) | N2—C20—H20B | 108.7 |
| C11—N2—C18 | 120.85 (11) | C21—C20—H20B | 108.7 |
| C20—N2—C18 | 117.79 (11) | H20A—C20—H20B | 107.6 |
| C28—N3—C29 | 122.51 (11) | C20—C21—H21A | 109.5 |
| C28—N3—C7 | 114.25 (10) | C20—C21—H21B | 109.5 |
| C29—N3—C7 | 123.22 (10) | H21A—C21—H21B | 109.5 |
| C31—N4—C30 | 115.75 (13) | C20—C21—H21C | 109.5 |
| O1—C1—C6 | 123.49 (11) | H21A—C21—H21C | 109.5 |
| O1—C1—C2 | 113.88 (11) | H21B—C21—H21C | 109.5 |
| C6—C1—C2 | 122.62 (12) | C23—C22—C27 | 120.78 (12) |
| C1—C2—C3 | 120.94 (12) | C23—C22—C7 | 128.55 (11) |
| C1—C2—H2 | 119.5 | C27—C22—C7 | 110.66 (11) |
| C3—C2—H2 | 119.5 | C22—C23—C24 | 117.82 (13) |

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| N1—C3—C2 | 120.63 (12) | C22—C23—H23 | 121.1 |
| N1—C3—C4 | 122.44 (12) | C24—C23—H23 | 121.1 |
| C2—C3—C4 | 116.92 (12) | C23—C24—C25 | 121.23 (13) |
| C5—C4—C3 | 120.51 (12) | C23—C24—H24 | 119.4 |
| C5—C4—H4A | 119.7 | C25—C24—H24 | 119.4 |
| C3—C4—H4A | 119.7 | C26—C25—C24 | 120.78 (13) |
| C4—C5—C6 | 122.88 (12) | C26—C25—H25 | 119.6 |
| C4—C5—H5 | 118.6 | C24—C25—H25 | 119.6 |
| C6—C5—H5 | 118.6 | C25—C26—C27 | 117.76 (12) |
| C1—C6—C5 | 116.09 (12) | C25—C26—H26 | 121.1 |
| C1—C6—C7 | 122.24 (11) | C27—C26—H26 | 121.1 |
| C5—C6—C7 | 121.63 (11) | C22—C27—C26 | 121.63 (12) |
| N3—C7—C8 | 111.39 (10) | C22—C27—C28 | 108.63 (11) |
| N3—C7—C6 | 110.37 (10) | C26—C27—C28 | 129.73 (12) |
| C8—C7—C6 | 110.12 (10) | O2—C28—N3 | 124.74 (12) |
| N3—C7—C22 | 99.66 (10) | O2—C28—C27 | 128.57 (12) |
| C8—C7—C22 | 112.68 (10) | N3—C28—C27 | 106.70 (11) |
| C6—C7—C22 | 112.25 (10) | N3—C29—C30 | 113.39 (11) |
| C13—C8—C9 | 115.62 (12) | N3—C29—H29A | 108.9 |
| C13—C8—C7 | 122.65 (11) | C30—C29—H29A | 108.9 |
| C9—C8—C7 | 121.73 (11) | N3—C29—H29B | 108.9 |
| C10—C9—C8 | 122.73 (12) | C30—C29—H29B | 108.9 |
| C10—C9—H9 | 118.6 | H29A—C29—H29B | 107.7 |
| C8—C9—H9 | 118.6 | N4—C30—C29 | 109.02 (12) |
| C9—C10—C11 | 120.95 (12) | N4—C30—H30A | 109.9 |
| C9—C10—H10 | 119.5 | C29—C30—H30A | 109.9 |
| C11—C10—H10 | 119.5 | N4—C30—H30B | 109.9 |
| N2—C11—C12 | 121.63 (12) | C29—C30—H30B | 109.9 |
| N2—C11—C10 | 121.41 (12) | H30A—C30—H30B | 108.3 |
| C12—C11—C10 | 116.95 (12) | N4—C31—C32 | 124.72 (14) |
| C13—C12—C11 | 120.57 (12) | N4—C31—H31 | 117.6 |
| C13—C12—H12 | 119.7 | C32—C31—H31 | 117.6 |
| C11—C12—H12 | 119.7 | C33—C32—C37 | 119.59 (13) |
| O1—C13—C8 | 123.02 (11) | C33—C32—C31 | 119.17 (13) |
| O1—C13—C12 | 113.79 (11) | C37—C32—C31 | 121.24 (13) |
| C8—C13—C12 | 123.18 (12) | C34—C33—C32 | 120.92 (13) |
| N1—C14—C15 | 113.99 (12) | C34—C33—H33 | 119.5 |
| N1—C14—H14A | 108.8 | C32—C33—H33 | 119.5 |
| C15—C14—H14A | 108.8 | C35—C34—C33 | 119.69 (13) |
| N1—C14—H14B | 108.8 | C35—C34—H34 | 120.2 |
| C15—C14—H14B | 108.8 | C33—C34—H34 | 120.2 |
| H14A—C14—H14B | 107.6 | O3—C35—C34 | 118.25 (13) |
| C14—C15—H15A | 109.5 | O3—C35—C36 | 121.73 (12) |
| C14—C15—H15B | 109.5 | C34—C35—C36 | 119.98 (13) |
| H15A—C15—H15B | 109.5 | O4—C36—C37 | 125.29 (13) |
| C14—C15—H15C | 109.5 | O4—C36—C35 | 114.45 (12) |
| H15A—C15—H15C | 109.5 | C37—C36—C35 | 120.26 (12) |
| H15B—C15—H15C | 109.5 | C36—C37—C32 | 119.53 (13) |

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| N1—C16—C17 | 113.44 (14) | C36—C37—H37 | 120.2 |
| N1—C16—H16A | 108.9 | C32—C37—H37 | 120.2 |
| C17—C16—H16A | 108.9 | O4—C38—H38A | 109.5 |
| N1—C16—H16B | 108.9 | O4—C38—H38B | 109.5 |
| C17—C16—H16B | 108.9 | H38A—C38—H38B | 109.5 |
| H16A—C16—H16B | 107.7 | O4—C38—H38C | 109.5 |
| C16—C17—H17A | 109.5 | H38A—C38—H38C | 109.5 |
| C16—C17—H17B | 109.5 | H38B—C38—H38C | 109.5 |
| H17A—C17—H17B | 109.5 | C39—O5—H5A | 109.5 |
| C16—C17—H17C | 109.5 | O5—C39—C40 | 109.53 (12) |
| H17A—C17—H17C | 109.5 | O5—C39—H39A | 109.8 |
| H17B—C17—H17C | 109.5 | C40—C39—H39A | 109.8 |
| N2—C18—C19 | 114.87 (12) | O5—C39—H39B | 109.8 |
| N2—C18—H18A | 108.6 | C40—C39—H39B | 109.8 |
| C19—C18—H18A | 108.6 | H39A—C39—H39B | 108.2 |
| N2—C18—H18B | 108.6 | C39—C40—H40A | 109.5 |
| C19—C18—H18B | 108.6 | C39—C40—H40B | 109.5 |
| H18A—C18—H18B | 107.5 | H40A—C40—H40B | 109.5 |
| C18—C19—H19A | 109.5 | C39—C40—H40C | 109.5 |
| C18—C19—H19B | 109.5 | H40A—C40—H40C | 109.5 |
| H19A—C19—H19B | 109.5 | H40B—C40—H40C | 109.5 |

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| C13—O1—C1—C6 | −4.83 (18) | C3—N1—C14—C15 | 89.30 (16) |
| C13—O1—C1—C2 | 176.30 (11) | C16—N1—C14—C15 | −94.71 (16) |
| O1—C1—C2—C3 | 177.85 (11) | C3—N1—C16—C17 | 88.72 (18) |
| C6—C1—C2—C3 | −1.0 (2) | C14—N1—C16—C17 | −87.22 (17) |
| C16—N1—C3—C2 | 169.76 (13) | C11—N2—C18—C19 | 83.16 (16) |
| C14—N1—C3—C2 | −14.5 (2) | C20—N2—C18—C19 | −89.23 (16) |
| C16—N1—C3—C4 | −11.1 (2) | C11—N2—C20—C21 | −76.12 (16) |
| C14—N1—C3—C4 | 164.66 (13) | C18—N2—C20—C21 | 96.26 (15) |
| C1—C2—C3—N1 | 178.44 (12) | N3—C7—C22—C23 | 177.55 (12) |
| C1—C2—C3—C4 | −0.7 (2) | C8—C7—C22—C23 | 59.38 (17) |
| N1—C3—C4—C5 | −177.39 (13) | C6—C7—C22—C23 | −65.64 (17) |
| C2—C3—C4—C5 | 1.8 (2) | N3—C7—C22—C27 | −1.82 (13) |
| C3—C4—C5—C6 | −1.1 (2) | C8—C7—C22—C27 | −119.99 (12) |
| O1—C1—C6—C5 | −177.09 (12) | C6—C7—C22—C27 | 114.99 (12) |
| C2—C1—C6—C5 | 1.68 (19) | C27—C22—C23—C24 | 0.62 (19) |
| O1—C1—C6—C7 | 0.63 (19) | C7—C22—C23—C24 | −178.69 (12) |
| C2—C1—C6—C7 | 179.41 (12) | C22—C23—C24—C25 | 0.0 (2) |
| C4—C5—C6—C1 | −0.6 (2) | C23—C24—C25—C26 | −0.7 (2) |
| C4—C5—C6—C7 | −178.35 (12) | C24—C25—C26—C27 | 0.7 (2) |
| C28—N3—C7—C8 | 122.41 (12) | C23—C22—C27—C26 | −0.62 (19) |
| C29—N3—C7—C8 | −59.14 (15) | C7—C22—C27—C26 | 178.81 (11) |
| C28—N3—C7—C6 | −114.94 (12) | C23—C22—C27—C28 | −179.42 (11) |
| C29—N3—C7—C6 | 63.51 (15) | C7—C22—C27—C28 | 0.01 (14) |
| C28—N3—C7—C22 | 3.28 (13) | C25—C26—C27—C22 | −0.06 (19) |
| C29—N3—C7—C22 | −178.27 (11) | C25—C26—C27—C28 | 178.47 (13) |
| C1—C6—C7—N3 | −119.49 (13) | C29—N3—C28—O2 | −1.7 (2) |

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| C5—C6—C7—N3 | 58.12 (15) | C7—N3—C28—O2 | 176.80 (12) |
| C1—C6—C7—C8 | 3.90 (16) | C29—N3—C28—C27 | 178.09 (11) |
| C5—C6—C7—C8 | −178.50 (11) | C7—N3—C28—C27 | −3.45 (14) |
| C1—C6—C7—C22 | 130.32 (13) | C22—C27—C28—O2 | −178.20 (13) |
| C5—C6—C7—C22 | −52.08 (16) | C26—C27—C28—O2 | 3.1 (2) |
| N3—C7—C8—C13 | 118.10 (13) | C22—C27—C28—N3 | 2.06 (14) |
| C6—C7—C8—C13 | −4.69 (16) | C26—C27—C28—N3 | −176.62 (13) |
| C22—C7—C8—C13 | −130.87 (12) | C28—N3—C29—C30 | 92.73 (15) |
| N3—C7—C8—C9 | −62.64 (15) | C7—N3—C29—C30 | −85.59 (15) |
| C6—C7—C8—C9 | 174.57 (11) | C31—N4—C30—C29 | 124.69 (14) |
| C22—C7—C8—C9 | 48.40 (16) | N3—C29—C30—N4 | −166.68 (12) |
| C13—C8—C9—C10 | 0.04 (19) | C30—N4—C31—C32 | −178.98 (12) |
| C7—C8—C9—C10 | −179.27 (12) | N4—C31—C32—C33 | −174.37 (14) |
| C8—C9—C10—C11 | −0.4 (2) | N4—C31—C32—C37 | 5.3 (2) |
| C20—N2—C11—C12 | 169.20 (12) | C37—C32—C33—C34 | −0.4 (2) |
| C18—N2—C11—C12 | −3.0 (2) | C31—C32—C33—C34 | 179.27 (12) |
| C20—N2—C11—C10 | −10.84 (19) | C32—C33—C34—C35 | −0.5 (2) |
| C18—N2—C11—C10 | 177.01 (12) | C33—C34—C35—O3 | 179.54 (12) |
| C9—C10—C11—N2 | −179.60 (12) | C33—C34—C35—C36 | 1.7 (2) |
| C9—C10—C11—C12 | 0.36 (19) | C38—O4—C36—C37 | 10.84 (19) |
| N2—C11—C12—C13 | −179.99 (12) | C38—O4—C36—C35 | −169.93 (12) |
| C10—C11—C12—C13 | 0.04 (19) | O3—C35—C36—O4 | 0.98 (18) |
| C1—O1—C13—C8 | 3.99 (18) | C34—C35—C36—O4 | 178.74 (12) |
| C1—O1—C13—C12 | −174.79 (11) | O3—C35—C36—C37 | −179.74 (12) |
| C9—C8—C13—O1 | −178.28 (12) | C34—C35—C36—C37 | −2.0 (2) |
| C7—C8—C13—O1 | 1.02 (19) | O4—C36—C37—C32 | −179.74 (12) |
| C9—C8—C13—C12 | 0.38 (19) | C35—C36—C37—C32 | 1.1 (2) |
| C7—C8—C13—C12 | 179.69 (12) | C33—C32—C37—C36 | 0.1 (2) |
| C11—C12—C13—O1 | 178.35 (11) | C31—C32—C37—C36 | −179.54 (12) |
| C11—C12—C13—C8 | −0.4 (2) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O3—H3···O5$^{i}$ | 0.84 | 1.96 | 2.7052 (15) | 148 |
| O5—H5*A*···O2 | 0.84 | 1.93 | 2.7552 (14) | 166 |

Symmetry code: (i) *x*+1/2, −*y*+3/2, *z*−1/2.