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Bis{6-[4-(2-ethoxyphenyldiazenyl)-phenoxy]hexanol} monohydrate

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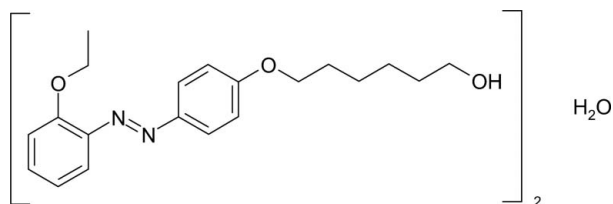
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 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å;
 R factor = 0.074; wR factor = 0.181; data-to-parameter ratio = 15.5.

The asymmetric unit of the title compound, $2\text{C}_{20}\text{H}_{26}\text{N}_2\text{O}_3 \cdot \text{H}_2\text{O}$, contains two independent molecules and one water molecule. The azo bonds adopt *trans* conformations and the dihedral angles between the aromatic rings in the two organic molecules are 4.5 (2) and 1.5 (2)°. In the crystal structure, $\text{O}-\text{H} \cdots \text{O}$ and $\text{C}-\text{H} \cdots \text{O}$ hydrogen bonds help to establish the packing.

Related literature

 For the synthesis, see: Zhao *et al.* (2002). For background, see: Bach *et al.* (1996).


Experimental

Crystal data

 $2\text{C}_{20}\text{H}_{26}\text{N}_2\text{O}_3 \cdot \text{H}_2\text{O}$
 $M_r = 702.87$
 Triclinic, $P\bar{1}$
 $a = 7.4586$ (7) Å

 $b = 11.5674$ (11) Å
 $c = 24.070$ (2) Å
 $\alpha = 90.46$ (3)°
 $\beta = 98.46$ (3)°

 $\gamma = 106.10$ (3)°
 $V = 1970.9$ (5) Å³
 $Z = 2$
 Mo $K\alpha$ radiation

 $\mu = 0.08$ mm⁻¹
 $T = 293$ (2) K
 $0.30 \times 0.20 \times 0.10$ mm

Data collection

 Enraf–Nonius CAD-4
 diffractometer
 Absorption correction: ψ scan
 (North *et al.*, 1968)
 $T_{\min} = 0.956$, $T_{\max} = 0.982$
 7726 measured reflections

 7134 independent reflections
 3403 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.077$
 3 standard reflections
 every 200 reflections
 intensity decay: 1%

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.074$
 $wR(F^2) = 0.181$
 $S = 1.00$
 7134 reflections

 460 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.21$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.30$ e Å⁻³
Table 1
 Hydrogen-bond geometry (Å, °).

| $D-H \cdots A$ | $D-H$ | $H \cdots A$ | $D \cdots A$ | $D-H \cdots A$ |
|---|-------|--------------|--------------|----------------|
| $\text{OW}-\text{HWB} \cdots \text{O4}$ | 0.85 | 2.32 | 2.713 (4) | 109 |
| $\text{OW}-\text{HWA} \cdots \text{O4}$ | 0.85 | 2.31 | 2.713 (4) | 110 |
| $\text{O1}-\text{H1A} \cdots \text{Ow}^i$ | 0.85 | 2.14 | 2.768 (4) | 130 |
| $\text{O4}-\text{H4C} \cdots \text{O1}^{ii}$ | 0.85 | 2.16 | 2.753 (4) | 127 |
| $\text{C15}-\text{H15A} \cdots \text{O2}^{iii}$ | 0.93 | 2.59 | 3.494 (5) | 166 |
| $\text{C35}-\text{H35A} \cdots \text{O5}^{iii}$ | 0.93 | 2.57 | 3.487 (5) | 170 |
| $\text{C40}-\text{H40C} \cdots \text{Ow}^{iv}$ | 0.96 | 2.59 | 3.332 (5) | 134 |

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

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); 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: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2877).

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supplementary materials

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Bis{6-[4-(2-ethoxyphenyldiazenyl)phenoxy]hexanol} monohydrate

R. Lu, M. Zhang, L. Han, B. Wang and H. Wang

Comment

The photophysical properties of azo compounds are of interest in the development of nonlinear optical and optical data storage materials (e.g. Bach *et al.*, 1996). As part of our studies in this area, we report herein the synthesis and crystal structure of the title compound, (I), (Fig. 1).

The title compound, C₂₀H₂₆N₂O₃, contains two independent molecules and one water molecule, which form intermolecular O—H...O and C—H...O hydrogen bonds (Table 1).

Experimental

The title compound was prepared by the literature method (Zhao *et al.*, 2002). Yellow blocks of (I) were obtained by slow evaporation of an ethanol solution.

Refinement

The H were placed geometrically with C—H = 0.93–0.97 Å and O—H = 0.85 Å and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C},\text{O})$ or $1.5U_{\text{eq}}(\text{methyl C})$.

Figures

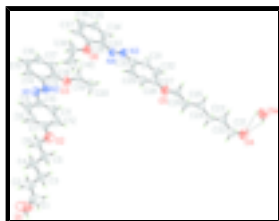


Fig. 1. A view of the molecular structure of (I), showing displacement ellipsoids at the 30% probability level. Dashed lines indicate hydrogen bonds.

Bis{6-[4-(2-ethoxyphenyldiazenyl)phenoxy]hexanol} monohydrate

Crystal data

2C₂₀H₂₆N₂O₃·H₂O

$M_r = 702.87$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 7.4586$ (7) Å

$b = 11.5674$ (11) Å

$Z = 2$

$F_{000} = 756$

$D_x = 1.184$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 25 reflections

$\theta = 9\text{--}13^\circ$

supplementary materials

| | |
|--------------------------------|---|
| $c = 24.070 (2) \text{ \AA}$ | $\mu = 0.08 \text{ mm}^{-1}$ |
| $\alpha = 90.46 (3)^\circ$ | $T = 293 \text{ K}$ |
| $\beta = 98.46 (3)^\circ$ | Block, yellow |
| $\gamma = 106.10 (3)^\circ$ | $0.30 \times 0.20 \times 0.10 \text{ mm}$ |
| $V = 1970.9 (5) \text{ \AA}^3$ | |

Data collection

| | |
|---|------------------------------------|
| Enraf-Nonius CAD-4 diffractometer | $R_{\text{int}} = 0.077$ |
| Radiation source: fine-focus sealed tube | $\theta_{\text{max}} = 25.3^\circ$ |
| Monochromator: graphite | $\theta_{\text{min}} = 1.7^\circ$ |
| $T = 293 \text{ K}$ | $h = 0 \rightarrow 8$ |
| $\omega/2\theta$ scans | $k = -13 \rightarrow 13$ |
| Absorption correction: ψ scan (North <i>et al.</i> , 1968) | $l = -28 \rightarrow 28$ |
| $T_{\text{min}} = 0.956$, $T_{\text{max}} = 0.982$ | 3 standard reflections |
| 7726 measured reflections | every 200 reflections |
| 7134 independent reflections | intensity decay: 1% |
| 3403 reflections with $I > 2\sigma(I)$ | |

Refinement

| | |
|--|--|
| Refinement on F^2 | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.074$ | H-atom parameters constrained |
| $wR(F^2) = 0.181$ | $w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + 1.3P]$ |
| $S = 1.00$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 7134 reflections | $(\Delta/\sigma)_{\text{max}} < 0.001$ |
| 460 parameters | $\Delta\rho_{\text{max}} = 0.21 \text{ e \AA}^{-3}$ |
| Primary atom site location: structure-invariant direct methods | $\Delta\rho_{\text{min}} = -0.30 \text{ e \AA}^{-3}$ |
| | Extinction correction: none |

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|-------------|-------------|---------------|----------------------------------|
| OW | 0.5276 (4) | 0.9282 (2) | -0.24533 (12) | 0.0971 (10) |
| HWB | 0.4522 | 0.8576 | -0.2505 | 0.117* |
| HWA | 0.4667 | 0.9734 | -0.2339 | 0.117* |
| O1 | -0.2094 (4) | 0.8215 (3) | 0.72415 (13) | 0.1066 (11) |
| H1A | -0.2390 | 0.8873 | 0.7249 | 0.128* |
| O2 | -0.2623 (4) | 0.2599 (2) | 0.47771 (11) | 0.0815 (8) |
| O3 | -0.2369 (4) | -0.2649 (2) | 0.26105 (10) | 0.0694 (7) |
| N1 | -0.2457 (5) | -0.2022 (3) | 0.41759 (13) | 0.0670 (9) |
| N2 | -0.2520 (4) | -0.2221 (2) | 0.36679 (13) | 0.0592 (8) |
| C1 | -0.2298 (6) | 0.7803 (4) | 0.66703 (19) | 0.0878 (14) |
| H1C | -0.1298 | 0.8320 | 0.6495 | 0.105* |
| H1D | -0.3495 | 0.7864 | 0.6472 | 0.105* |
| C2 | -0.2224 (6) | 0.6527 (3) | 0.66147 (16) | 0.0724 (11) |
| H2B | -0.3242 | 0.6014 | 0.6784 | 0.087* |
| H2C | -0.1043 | 0.6467 | 0.6825 | 0.087* |
| C3 | -0.2381 (6) | 0.6057 (3) | 0.60160 (16) | 0.0725 (11) |
| H3A | -0.1302 | 0.6523 | 0.5856 | 0.087* |
| H3B | -0.3509 | 0.6176 | 0.5796 | 0.087* |
| C4 | -0.2468 (5) | 0.4737 (3) | 0.59679 (15) | 0.0639 (10) |
| H4A | -0.3572 | 0.4271 | 0.6117 | 0.077* |
| H4B | -0.1363 | 0.4615 | 0.6200 | 0.077* |
| C5 | -0.2556 (6) | 0.4259 (3) | 0.53730 (15) | 0.0674 (10) |
| H5A | -0.3703 | 0.4327 | 0.5144 | 0.081* |
| H5B | -0.1490 | 0.4747 | 0.5214 | 0.081* |
| C6 | -0.2523 (6) | 0.2965 (3) | 0.53547 (15) | 0.0682 (11) |
| H6A | -0.1369 | 0.2886 | 0.5575 | 0.082* |
| H6B | -0.3589 | 0.2464 | 0.5509 | 0.082* |
| C7 | -0.2574 (5) | 0.1447 (3) | 0.46608 (16) | 0.0641 (10) |
| C8 | -0.2501 (6) | 0.0598 (3) | 0.50434 (16) | 0.0780 (12) |
| H8A | -0.2476 | 0.0779 | 0.5422 | 0.094* |
| C9 | -0.2466 (6) | -0.0536 (4) | 0.48678 (16) | 0.0810 (13) |
| H9A | -0.2415 | -0.1114 | 0.5131 | 0.097* |
| C10 | -0.2504 (5) | -0.0821 (3) | 0.43104 (15) | 0.0578 (9) |
| C11 | -0.2537 (6) | 0.0043 (3) | 0.39299 (16) | 0.0712 (11) |
| H11A | -0.2522 | -0.0129 | 0.3553 | 0.085* |
| C12 | -0.2592 (6) | 0.1153 (4) | 0.40993 (16) | 0.0804 (13) |
| H12A | -0.2643 | 0.1728 | 0.3834 | 0.096* |
| C13 | -0.2518 (5) | -0.3409 (3) | 0.35068 (15) | 0.0527 (9) |
| C14 | -0.2609 (5) | -0.4337 (3) | 0.38626 (16) | 0.0648 (10) |
| H14A | -0.2637 | -0.4199 | 0.4242 | 0.078* |
| C15 | -0.2659 (5) | -0.5478 (3) | 0.36662 (18) | 0.0702 (11) |
| H15A | -0.2720 | -0.6102 | 0.3910 | 0.084* |
| C16 | -0.2618 (5) | -0.5669 (4) | 0.31014 (19) | 0.0725 (11) |
| H16A | -0.2657 | -0.6431 | 0.2965 | 0.087* |
| C17 | -0.2520 (5) | -0.4757 (3) | 0.27399 (17) | 0.0656 (10) |

supplementary materials

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|------|-------------|-------------|---------------|-------------|
| H17A | -0.2481 | -0.4901 | 0.2362 | 0.079* |
| C18 | -0.2479 (5) | -0.3613 (3) | 0.29358 (15) | 0.0556 (9) |
| C19 | -0.2130 (6) | -0.2753 (3) | 0.20418 (15) | 0.0675 (11) |
| H19A | -0.3237 | -0.3317 | 0.1829 | 0.081* |
| H19B | -0.1040 | -0.3042 | 0.2016 | 0.081* |
| C20 | -0.1848 (6) | -0.1522 (4) | 0.18134 (17) | 0.0864 (13) |
| H20A | -0.1668 | -0.1557 | 0.1428 | 0.130* |
| H20B | -0.0755 | -0.0972 | 0.2029 | 0.130* |
| H20C | -0.2940 | -0.1250 | 0.1838 | 0.130* |
| O4 | 0.1625 (4) | 0.8614 (2) | -0.22791 (12) | 0.0906 (9) |
| H4C | 0.0704 | 0.8070 | -0.2458 | 0.109* |
| O5 | 0.2453 (4) | 0.3308 (2) | 0.01659 (10) | 0.0768 (8) |
| O6 | 0.2785 (4) | -0.1956 (2) | 0.24180 (10) | 0.0668 (7) |
| N3 | 0.2699 (5) | -0.1229 (3) | 0.08391 (13) | 0.0689 (9) |
| N4 | 0.2798 (4) | -0.1396 (2) | 0.13469 (12) | 0.0581 (8) |
| C21 | 0.1799 (6) | 0.8282 (4) | -0.17127 (19) | 0.0852 (13) |
| H21A | 0.0667 | 0.8301 | -0.1564 | 0.102* |
| H21B | 0.2857 | 0.8874 | -0.1494 | 0.102* |
| C22 | 0.2088 (6) | 0.7064 (3) | -0.16406 (16) | 0.0707 (11) |
| H22A | 0.3261 | 0.7065 | -0.1769 | 0.085* |
| H22B | 0.1073 | 0.6484 | -0.1881 | 0.085* |
| C23 | 0.2158 (6) | 0.6645 (3) | -0.10461 (16) | 0.0661 (10) |
| H23A | 0.3228 | 0.7186 | -0.0808 | 0.079* |
| H23B | 0.1020 | 0.6683 | -0.0906 | 0.079* |
| C24 | 0.2325 (6) | 0.5374 (3) | -0.10095 (15) | 0.0658 (10) |
| H24A | 0.1279 | 0.4842 | -0.1260 | 0.079* |
| H24B | 0.3484 | 0.5346 | -0.1139 | 0.079* |
| C25 | 0.2330 (6) | 0.4913 (3) | -0.04241 (15) | 0.0664 (10) |
| H25A | 0.3431 | 0.5407 | -0.0179 | 0.080* |
| H25B | 0.1217 | 0.4995 | -0.0282 | 0.080* |
| C26 | 0.2352 (6) | 0.3623 (3) | -0.04032 (15) | 0.0671 (10) |
| H26A | 0.1215 | 0.3109 | -0.0626 | 0.081* |
| H26B | 0.3435 | 0.3519 | -0.0555 | 0.081* |
| C27 | 0.2510 (5) | 0.2175 (3) | 0.03082 (15) | 0.0614 (10) |
| C28 | 0.2504 (6) | 0.1940 (3) | 0.08671 (16) | 0.0737 (12) |
| H28A | 0.2452 | 0.2539 | 0.1119 | 0.088* |
| C29 | 0.2573 (6) | 0.0849 (3) | 0.10573 (15) | 0.0677 (11) |
| H29A | 0.2566 | 0.0707 | 0.1437 | 0.081* |
| C30 | 0.2656 (5) | -0.0058 (3) | 0.06852 (15) | 0.0616 (10) |
| C31 | 0.2649 (7) | 0.0181 (4) | 0.01333 (16) | 0.0855 (14) |
| H31A | 0.2687 | -0.0422 | -0.0119 | 0.103* |
| C32 | 0.2589 (7) | 0.1291 (4) | -0.00667 (17) | 0.0846 (13) |
| H32A | 0.2601 | 0.1436 | -0.0446 | 0.102* |
| C33 | 0.2824 (5) | -0.2583 (3) | 0.14889 (15) | 0.0536 (9) |
| C34 | 0.2858 (5) | -0.3467 (3) | 0.11001 (16) | 0.0619 (10) |
| H34A | 0.2862 | -0.3292 | 0.0724 | 0.074* |
| C35 | 0.2887 (5) | -0.4604 (3) | 0.12658 (19) | 0.0705 (11) |
| H35A | 0.2898 | -0.5191 | 0.1001 | 0.085* |
| C36 | 0.2898 (5) | -0.4861 (3) | 0.1814 (2) | 0.0741 (11) |

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|------|------------|-------------|--------------|-------------|
| H36A | 0.2926 | -0.5627 | 0.1922 | 0.089* |
| C37 | 0.2870 (5) | -0.4001 (3) | 0.22214 (17) | 0.0679 (11) |
| H37A | 0.2871 | -0.4187 | 0.2597 | 0.081* |
| C38 | 0.2839 (5) | -0.2860 (3) | 0.20512 (16) | 0.0580 (9) |
| C39 | 0.2613 (6) | -0.2236 (4) | 0.29842 (15) | 0.0755 (12) |
| H39A | 0.3771 | -0.2374 | 0.3173 | 0.091* |
| H39B | 0.1586 | -0.2960 | 0.2996 | 0.091* |
| C40 | 0.2227 (7) | -0.1189 (4) | 0.32730 (17) | 0.0897 (14) |
| H40A | 0.2108 | -0.1361 | 0.3658 | 0.135* |
| H40B | 0.1075 | -0.1062 | 0.3085 | 0.135* |
| H40C | 0.3252 | -0.0477 | 0.3261 | 0.135* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|-------------|--------------|--------------|
| OW | 0.102 (2) | 0.0739 (19) | 0.120 (2) | 0.0148 (17) | 0.0522 (19) | -0.0047 (17) |
| O1 | 0.111 (3) | 0.095 (2) | 0.123 (3) | 0.0508 (19) | 0.008 (2) | -0.050 (2) |
| O2 | 0.128 (3) | 0.0624 (17) | 0.0648 (18) | 0.0391 (17) | 0.0244 (16) | -0.0029 (13) |
| O3 | 0.097 (2) | 0.0609 (16) | 0.0555 (16) | 0.0248 (15) | 0.0232 (14) | -0.0032 (13) |
| N1 | 0.089 (2) | 0.062 (2) | 0.055 (2) | 0.0276 (18) | 0.0165 (17) | -0.0053 (15) |
| N2 | 0.066 (2) | 0.0560 (19) | 0.059 (2) | 0.0185 (15) | 0.0193 (16) | -0.0019 (15) |
| C1 | 0.091 (3) | 0.073 (3) | 0.102 (4) | 0.031 (3) | 0.010 (3) | -0.029 (3) |
| C2 | 0.065 (3) | 0.073 (3) | 0.081 (3) | 0.026 (2) | 0.006 (2) | -0.019 (2) |
| C3 | 0.083 (3) | 0.057 (2) | 0.081 (3) | 0.021 (2) | 0.025 (2) | -0.011 (2) |
| C4 | 0.065 (3) | 0.060 (2) | 0.072 (3) | 0.0217 (19) | 0.018 (2) | -0.0085 (19) |
| C5 | 0.083 (3) | 0.061 (2) | 0.065 (2) | 0.029 (2) | 0.015 (2) | -0.0009 (19) |
| C6 | 0.087 (3) | 0.066 (3) | 0.055 (2) | 0.024 (2) | 0.015 (2) | -0.0097 (19) |
| C7 | 0.078 (3) | 0.052 (2) | 0.067 (3) | 0.022 (2) | 0.020 (2) | -0.008 (2) |
| C8 | 0.125 (4) | 0.066 (3) | 0.052 (2) | 0.040 (3) | 0.017 (2) | -0.002 (2) |
| C9 | 0.130 (4) | 0.068 (3) | 0.053 (2) | 0.038 (3) | 0.021 (2) | 0.004 (2) |
| C10 | 0.063 (2) | 0.050 (2) | 0.063 (2) | 0.0184 (18) | 0.0158 (19) | -0.0029 (18) |
| C11 | 0.101 (3) | 0.063 (3) | 0.055 (2) | 0.026 (2) | 0.024 (2) | -0.004 (2) |
| C12 | 0.129 (4) | 0.064 (3) | 0.057 (3) | 0.036 (3) | 0.027 (2) | 0.002 (2) |
| C13 | 0.048 (2) | 0.050 (2) | 0.063 (2) | 0.0176 (17) | 0.0127 (17) | -0.0029 (18) |
| C14 | 0.071 (3) | 0.058 (2) | 0.071 (3) | 0.022 (2) | 0.020 (2) | 0.003 (2) |
| C15 | 0.071 (3) | 0.057 (2) | 0.086 (3) | 0.022 (2) | 0.017 (2) | 0.011 (2) |
| C16 | 0.073 (3) | 0.057 (3) | 0.091 (3) | 0.023 (2) | 0.015 (2) | -0.005 (2) |
| C17 | 0.072 (3) | 0.057 (2) | 0.070 (3) | 0.021 (2) | 0.013 (2) | -0.010 (2) |
| C18 | 0.057 (2) | 0.052 (2) | 0.058 (2) | 0.0153 (18) | 0.0094 (18) | 0.0027 (18) |
| C19 | 0.066 (3) | 0.075 (3) | 0.061 (2) | 0.016 (2) | 0.018 (2) | -0.006 (2) |
| C20 | 0.100 (4) | 0.084 (3) | 0.073 (3) | 0.015 (3) | 0.026 (3) | 0.009 (2) |
| O4 | 0.089 (2) | 0.082 (2) | 0.091 (2) | 0.0152 (17) | -0.0022 (17) | 0.0206 (17) |
| O5 | 0.114 (2) | 0.0645 (17) | 0.0610 (17) | 0.0322 (16) | 0.0270 (15) | 0.0108 (13) |
| O6 | 0.0854 (19) | 0.0619 (16) | 0.0582 (16) | 0.0240 (14) | 0.0210 (13) | 0.0053 (13) |
| N3 | 0.100 (3) | 0.060 (2) | 0.053 (2) | 0.0308 (18) | 0.0152 (18) | 0.0057 (15) |
| N4 | 0.065 (2) | 0.0559 (19) | 0.057 (2) | 0.0197 (16) | 0.0174 (15) | 0.0067 (15) |
| C21 | 0.090 (3) | 0.078 (3) | 0.099 (4) | 0.032 (3) | 0.037 (3) | 0.024 (3) |
| C22 | 0.081 (3) | 0.062 (2) | 0.070 (3) | 0.019 (2) | 0.019 (2) | 0.012 (2) |

supplementary materials

| | | | | | | |
|-----|-----------|-----------|-----------|-------------|-------------|--------------|
| C23 | 0.071 (3) | 0.059 (2) | 0.075 (3) | 0.023 (2) | 0.020 (2) | 0.0070 (19) |
| C24 | 0.075 (3) | 0.063 (2) | 0.069 (3) | 0.027 (2) | 0.024 (2) | 0.0096 (19) |
| C25 | 0.075 (3) | 0.064 (2) | 0.068 (3) | 0.029 (2) | 0.017 (2) | 0.0091 (19) |
| C26 | 0.076 (3) | 0.069 (3) | 0.060 (2) | 0.023 (2) | 0.019 (2) | 0.009 (2) |
| C27 | 0.074 (3) | 0.055 (2) | 0.059 (2) | 0.023 (2) | 0.013 (2) | 0.0095 (19) |
| C28 | 0.115 (4) | 0.057 (2) | 0.060 (3) | 0.032 (2) | 0.031 (2) | 0.007 (2) |
| C29 | 0.093 (3) | 0.070 (3) | 0.047 (2) | 0.028 (2) | 0.024 (2) | 0.0092 (19) |
| C30 | 0.074 (3) | 0.057 (2) | 0.059 (2) | 0.023 (2) | 0.018 (2) | 0.0036 (18) |
| C31 | 0.151 (4) | 0.069 (3) | 0.051 (2) | 0.052 (3) | 0.021 (3) | 0.001 (2) |
| C32 | 0.139 (4) | 0.072 (3) | 0.055 (2) | 0.043 (3) | 0.029 (3) | 0.005 (2) |
| C33 | 0.054 (2) | 0.047 (2) | 0.063 (2) | 0.0159 (17) | 0.0123 (18) | 0.0005 (17) |
| C34 | 0.062 (3) | 0.062 (2) | 0.067 (2) | 0.023 (2) | 0.0120 (19) | -0.0024 (19) |
| C35 | 0.066 (3) | 0.058 (3) | 0.091 (3) | 0.024 (2) | 0.011 (2) | -0.009 (2) |
| C36 | 0.068 (3) | 0.051 (2) | 0.104 (3) | 0.018 (2) | 0.014 (2) | 0.008 (2) |
| C37 | 0.065 (3) | 0.060 (3) | 0.080 (3) | 0.020 (2) | 0.012 (2) | 0.011 (2) |
| C38 | 0.053 (2) | 0.053 (2) | 0.070 (3) | 0.0146 (18) | 0.0137 (19) | -0.0017 (19) |
| C39 | 0.084 (3) | 0.086 (3) | 0.056 (3) | 0.022 (2) | 0.015 (2) | 0.015 (2) |
| C40 | 0.112 (4) | 0.095 (3) | 0.059 (3) | 0.016 (3) | 0.027 (2) | -0.003 (2) |

Geometric parameters (Å, °)

| | | | |
|--------|-----------|----------|-----------|
| OW—HWB | 0.8500 | C20—H20C | 0.9600 |
| OW—HWA | 0.8500 | O4—C21 | 1.417 (4) |
| O1—C1 | 1.424 (5) | O4—H4C | 0.8501 |
| O1—H1A | 0.8499 | O5—C27 | 1.368 (4) |
| O2—C7 | 1.371 (4) | O5—C26 | 1.417 (4) |
| O2—C6 | 1.436 (4) | O6—C38 | 1.376 (4) |
| O3—C18 | 1.359 (4) | O6—C39 | 1.419 (4) |
| O3—C19 | 1.415 (4) | N3—N4 | 1.234 (4) |
| N1—N2 | 1.234 (4) | N3—C30 | 1.414 (4) |
| N1—C10 | 1.434 (4) | N4—C33 | 1.422 (4) |
| N2—C13 | 1.425 (4) | C21—C22 | 1.492 (5) |
| C1—C2 | 1.497 (5) | C21—H21A | 0.9700 |
| C1—H1C | 0.9700 | C21—H21B | 0.9700 |
| C1—H1D | 0.9700 | C22—C23 | 1.513 (5) |
| C2—C3 | 1.513 (5) | C22—H22A | 0.9700 |
| C2—H2B | 0.9700 | C22—H22B | 0.9700 |
| C2—H2C | 0.9700 | C23—C24 | 1.512 (4) |
| C3—C4 | 1.513 (5) | C23—H23A | 0.9700 |
| C3—H3A | 0.9700 | C23—H23B | 0.9700 |
| C3—H3B | 0.9700 | C24—C25 | 1.511 (5) |
| C4—C5 | 1.516 (5) | C24—H24A | 0.9700 |
| C4—H4A | 0.9700 | C24—H24B | 0.9700 |
| C4—H4B | 0.9700 | C25—C26 | 1.499 (5) |
| C5—C6 | 1.504 (5) | C25—H25A | 0.9700 |
| C5—H5A | 0.9700 | C25—H25B | 0.9700 |
| C5—H5B | 0.9700 | C26—H26A | 0.9700 |
| C6—H6A | 0.9700 | C26—H26B | 0.9700 |
| C6—H6B | 0.9700 | C27—C28 | 1.375 (5) |

| | | | |
|------------|-----------|---------------|-----------|
| C7—C8 | 1.358 (5) | C27—C32 | 1.378 (5) |
| C7—C12 | 1.388 (5) | C28—C29 | 1.357 (5) |
| C8—C9 | 1.383 (5) | C28—H28A | 0.9300 |
| C8—H8A | 0.9300 | C29—C30 | 1.394 (5) |
| C9—C10 | 1.373 (5) | C29—H29A | 0.9300 |
| C9—H9A | 0.9300 | C30—C31 | 1.359 (5) |
| C10—C11 | 1.364 (5) | C31—C32 | 1.384 (5) |
| C11—C12 | 1.357 (5) | C31—H31A | 0.9300 |
| C11—H11A | 0.9300 | C32—H32A | 0.9300 |
| C12—H12A | 0.9300 | C33—C34 | 1.389 (4) |
| C13—C14 | 1.372 (5) | C33—C38 | 1.393 (5) |
| C13—C18 | 1.399 (5) | C34—C35 | 1.382 (5) |
| C14—C15 | 1.387 (5) | C34—H34A | 0.9300 |
| C14—H14A | 0.9300 | C35—C36 | 1.355 (5) |
| C15—C16 | 1.382 (5) | C35—H35A | 0.9300 |
| C15—H15A | 0.9300 | C36—C37 | 1.397 (5) |
| C16—C17 | 1.365 (5) | C36—H36A | 0.9300 |
| C16—H16A | 0.9300 | C37—C38 | 1.390 (5) |
| C17—C18 | 1.392 (5) | C37—H37A | 0.9300 |
| C17—H17A | 0.9300 | C39—C40 | 1.507 (5) |
| C19—C20 | 1.501 (5) | C39—H39A | 0.9700 |
| C19—H19A | 0.9700 | C39—H39B | 0.9700 |
| C19—H19B | 0.9700 | C40—H40A | 0.9600 |
| C20—H20A | 0.9600 | C40—H40B | 0.9600 |
| C20—H20B | 0.9600 | C40—H40C | 0.9600 |
| HWB—OW—HWA | 106.9 | C21—O4—H4C | 106.1 |
| C1—O1—H1A | 108.6 | C27—O5—C26 | 120.5 (3) |
| C7—O2—C6 | 117.8 (3) | C38—O6—C39 | 118.1 (3) |
| C18—O3—C19 | 119.5 (3) | N4—N3—C30 | 115.4 (3) |
| N2—N1—C10 | 112.9 (3) | N3—N4—C33 | 114.2 (3) |
| N1—N2—C13 | 115.5 (3) | O4—C21—C22 | 113.7 (4) |
| O1—C1—C2 | 112.5 (4) | O4—C21—H21A | 108.8 |
| O1—C1—H1C | 109.1 | C22—C21—H21A | 108.8 |
| C2—C1—H1C | 109.1 | O4—C21—H21B | 108.8 |
| O1—C1—H1D | 109.1 | C22—C21—H21B | 108.8 |
| C2—C1—H1D | 109.1 | H21A—C21—H21B | 107.7 |
| H1C—C1—H1D | 107.8 | C21—C22—C23 | 115.2 (3) |
| C1—C2—C3 | 114.5 (3) | C21—C22—H22A | 108.5 |
| C1—C2—H2B | 108.6 | C23—C22—H22A | 108.5 |
| C3—C2—H2B | 108.6 | C21—C22—H22B | 108.5 |
| C1—C2—H2C | 108.6 | C23—C22—H22B | 108.5 |
| C3—C2—H2C | 108.6 | H22A—C22—H22B | 107.5 |
| H2B—C2—H2C | 107.6 | C24—C23—C22 | 112.4 (3) |
| C2—C3—C4 | 113.5 (3) | C24—C23—H23A | 109.1 |
| C2—C3—H3A | 108.9 | C22—C23—H23A | 109.1 |
| C4—C3—H3A | 108.9 | C24—C23—H23B | 109.1 |
| C2—C3—H3B | 108.9 | C22—C23—H23B | 109.1 |
| C4—C3—H3B | 108.9 | H23A—C23—H23B | 107.9 |
| H3A—C3—H3B | 107.7 | C25—C24—C23 | 113.7 (3) |

supplementary materials

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|--------------|-----------|---------------|-----------|
| C3—C4—C5 | 114.3 (3) | C25—C24—H24A | 108.8 |
| C3—C4—H4A | 108.7 | C23—C24—H24A | 108.8 |
| C5—C4—H4A | 108.7 | C25—C24—H24B | 108.8 |
| C3—C4—H4B | 108.7 | C23—C24—H24B | 108.8 |
| C5—C4—H4B | 108.7 | H24A—C24—H24B | 107.7 |
| H4A—C4—H4B | 107.6 | C26—C25—C24 | 113.0 (3) |
| C6—C5—C4 | 111.9 (3) | C26—C25—H25A | 109.0 |
| C6—C5—H5A | 109.2 | C24—C25—H25A | 109.0 |
| C4—C5—H5A | 109.2 | C26—C25—H25B | 109.0 |
| C6—C5—H5B | 109.2 | C24—C25—H25B | 109.0 |
| C4—C5—H5B | 109.2 | H25A—C25—H25B | 107.8 |
| H5A—C5—H5B | 107.9 | O5—C26—C25 | 108.1 (3) |
| O2—C6—C5 | 107.6 (3) | O5—C26—H26A | 110.1 |
| O2—C6—H6A | 110.2 | C25—C26—H26A | 110.1 |
| C5—C6—H6A | 110.2 | O5—C26—H26B | 110.1 |
| O2—C6—H6B | 110.2 | C25—C26—H26B | 110.1 |
| C5—C6—H6B | 110.2 | H26A—C26—H26B | 108.4 |
| H6A—C6—H6B | 108.5 | O5—C27—C28 | 116.0 (3) |
| C8—C7—O2 | 125.7 (3) | O5—C27—C32 | 124.4 (3) |
| C8—C7—C12 | 118.7 (3) | C28—C27—C32 | 119.6 (3) |
| O2—C7—C12 | 115.6 (3) | C29—C28—C27 | 121.1 (4) |
| C7—C8—C9 | 119.8 (4) | C29—C28—H28A | 119.4 |
| C7—C8—H8A | 120.1 | C27—C28—H28A | 119.4 |
| C9—C8—H8A | 120.1 | C28—C29—C30 | 120.2 (3) |
| C10—C9—C8 | 121.0 (4) | C28—C29—H29A | 119.9 |
| C10—C9—H9A | 119.5 | C30—C29—H29A | 119.9 |
| C8—C9—H9A | 119.5 | C31—C30—C29 | 118.2 (3) |
| C11—C10—C9 | 118.9 (3) | C31—C30—N3 | 117.2 (3) |
| C11—C10—N1 | 124.8 (3) | C29—C30—N3 | 124.6 (3) |
| C9—C10—N1 | 116.3 (3) | C30—C31—C32 | 122.3 (4) |
| C12—C11—C10 | 120.3 (4) | C30—C31—H31A | 118.8 |
| C12—C11—H11A | 119.9 | C32—C31—H31A | 118.8 |
| C10—C11—H11A | 119.9 | C27—C32—C31 | 118.5 (4) |
| C11—C12—C7 | 121.3 (4) | C27—C32—H32A | 120.7 |
| C11—C12—H12A | 119.4 | C31—C32—H32A | 120.7 |
| C7—C12—H12A | 119.4 | C34—C33—C38 | 118.6 (3) |
| C14—C13—C18 | 119.6 (3) | C34—C33—N4 | 123.6 (3) |
| C14—C13—N2 | 124.9 (3) | C38—C33—N4 | 117.7 (3) |
| C18—C13—N2 | 115.4 (3) | C35—C34—C33 | 120.8 (4) |
| C13—C14—C15 | 121.1 (4) | C35—C34—H34A | 119.6 |
| C13—C14—H14A | 119.4 | C33—C34—H34A | 119.6 |
| C15—C14—H14A | 119.4 | C36—C35—C34 | 119.9 (4) |
| C16—C15—C14 | 118.7 (4) | C36—C35—H35A | 120.1 |
| C16—C15—H15A | 120.6 | C34—C35—H35A | 120.1 |
| C14—C15—H15A | 120.6 | C35—C36—C37 | 121.5 (4) |
| C17—C16—C15 | 121.2 (4) | C35—C36—H36A | 119.3 |
| C17—C16—H16A | 119.4 | C37—C36—H36A | 119.3 |
| C15—C16—H16A | 119.4 | C38—C37—C36 | 118.3 (4) |
| C16—C17—C18 | 120.2 (4) | C38—C37—H37A | 120.9 |

| | | | |
|-----------------|------------|-----------------|------------|
| C16—C17—H17A | 119.9 | C36—C37—H37A | 120.9 |
| C18—C17—H17A | 119.9 | O6—C38—C37 | 122.8 (3) |
| O3—C18—C17 | 124.5 (3) | O6—C38—C33 | 116.3 (3) |
| O3—C18—C13 | 116.3 (3) | C37—C38—C33 | 120.9 (3) |
| C17—C18—C13 | 119.2 (3) | O6—C39—C40 | 108.2 (3) |
| O3—C19—C20 | 107.2 (3) | O6—C39—H39A | 110.1 |
| O3—C19—H19A | 110.3 | C40—C39—H39A | 110.1 |
| C20—C19—H19A | 110.3 | O6—C39—H39B | 110.1 |
| O3—C19—H19B | 110.3 | C40—C39—H39B | 110.1 |
| C20—C19—H19B | 110.3 | H39A—C39—H39B | 108.4 |
| H19A—C19—H19B | 108.5 | C39—C40—H40A | 109.5 |
| C19—C20—H20A | 109.5 | C39—C40—H40B | 109.5 |
| C19—C20—H20B | 109.5 | H40A—C40—H40B | 109.5 |
| H20A—C20—H20B | 109.5 | C39—C40—H40C | 109.5 |
| C19—C20—H20C | 109.5 | H40A—C40—H40C | 109.5 |
| H20A—C20—H20C | 109.5 | H40B—C40—H40C | 109.5 |
| H20B—C20—H20C | 109.5 | | |
| C10—N1—N2—C13 | -178.7 (3) | C30—N3—N4—C33 | 179.5 (3) |
| O1—C1—C2—C3 | -178.4 (3) | O4—C21—C22—C23 | -176.3 (3) |
| C1—C2—C3—C4 | -175.2 (3) | C21—C22—C23—C24 | 176.3 (4) |
| C2—C3—C4—C5 | -178.0 (3) | C22—C23—C24—C25 | -178.0 (3) |
| C3—C4—C5—C6 | 176.6 (3) | C23—C24—C25—C26 | 175.7 (3) |
| C7—O2—C6—C5 | 178.7 (3) | C27—O5—C26—C25 | -179.4 (3) |
| C4—C5—C6—O2 | 179.6 (3) | C24—C25—C26—O5 | 177.0 (3) |
| C6—O2—C7—C8 | 2.4 (6) | C26—O5—C27—C28 | -176.6 (3) |
| C6—O2—C7—C12 | -177.3 (4) | C26—O5—C27—C32 | 4.0 (6) |
| O2—C7—C8—C9 | 179.7 (4) | O5—C27—C28—C29 | -179.4 (4) |
| C12—C7—C8—C9 | -0.6 (7) | C32—C27—C28—C29 | -0.1 (6) |
| C7—C8—C9—C10 | 0.0 (7) | C27—C28—C29—C30 | 0.1 (6) |
| C8—C9—C10—C11 | 1.4 (6) | C28—C29—C30—C31 | -0.5 (6) |
| C8—C9—C10—N1 | 179.9 (4) | C28—C29—C30—N3 | -178.9 (4) |
| N2—N1—C10—C11 | -2.5 (5) | N4—N3—C30—C31 | 176.6 (4) |
| N2—N1—C10—C9 | 179.1 (4) | N4—N3—C30—C29 | -5.1 (6) |
| C9—C10—C11—C12 | -2.1 (6) | C29—C30—C31—C32 | 0.8 (7) |
| N1—C10—C11—C12 | 179.6 (4) | N3—C30—C31—C32 | 179.3 (4) |
| C10—C11—C12—C7 | 1.4 (7) | O5—C27—C32—C31 | 179.7 (4) |
| C8—C7—C12—C11 | 0.0 (7) | C28—C27—C32—C31 | 0.4 (6) |
| O2—C7—C12—C11 | 179.6 (4) | C30—C31—C32—C27 | -0.8 (7) |
| N1—N2—C13—C14 | 5.6 (5) | N3—N4—C33—C34 | 4.0 (5) |
| N1—N2—C13—C18 | -176.5 (3) | N3—N4—C33—C38 | -176.9 (3) |
| C18—C13—C14—C15 | 0.1 (5) | C38—C33—C34—C35 | 0.7 (5) |
| N2—C13—C14—C15 | 177.9 (3) | N4—C33—C34—C35 | 179.8 (3) |
| C13—C14—C15—C16 | 0.0 (6) | C33—C34—C35—C36 | -0.6 (6) |
| C14—C15—C16—C17 | 0.3 (6) | C34—C35—C36—C37 | 0.4 (6) |
| C15—C16—C17—C18 | -0.6 (6) | C35—C36—C37—C38 | -0.3 (6) |
| C19—O3—C18—C17 | -5.3 (5) | C39—O6—C38—C37 | -4.8 (5) |
| C19—O3—C18—C13 | 173.6 (3) | C39—O6—C38—C33 | 174.0 (3) |
| C16—C17—C18—O3 | 179.5 (3) | C36—C37—C38—O6 | 179.2 (3) |
| C16—C17—C18—C13 | 0.7 (5) | C36—C37—C38—C33 | 0.4 (5) |

supplementary materials

| | | | |
|-----------------|------------|-----------------|------------|
| C14—C13—C18—O3 | -179.4 (3) | C34—C33—C38—O6 | -179.4 (3) |
| N2—C13—C18—O3 | 2.6 (4) | N4—C33—C38—O6 | 1.4 (5) |
| C14—C13—C18—C17 | -0.4 (5) | C34—C33—C38—C37 | -0.6 (5) |
| N2—C13—C18—C17 | -178.5 (3) | N4—C33—C38—C37 | -179.8 (3) |
| C18—O3—C19—C20 | -173.9 (3) | C38—O6—C39—C40 | -169.8 (3) |

Hydrogen-bond geometry (\AA , $^\circ$)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|-------------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| OW—HWB \cdots O4 | 0.85 | 2.32 | 2.713 (4) | 109 |
| OW—HWA \cdots O4 | 0.85 | 2.31 | 2.713 (4) | 110 |
| O1—H1A \cdots OW ⁱ | 0.85 | 2.14 | 2.768 (4) | 130 |
| O4—H4C \cdots O1 ⁱⁱ | 0.85 | 2.16 | 2.753 (4) | 127 |
| C15—H15A \cdots O2 ⁱⁱⁱ | 0.93 | 2.59 | 3.494 (5) | 166 |
| C35—H35A \cdots O5 ⁱⁱⁱ | 0.93 | 2.57 | 3.487 (5) | 170 |
| C40—H40C \cdots OW ^{iv} | 0.96 | 2.59 | 3.332 (5) | 134 |

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

Fig. 1

