Crystal structure and Hirshfeld surface analysis of 1-(tert-butylamino)-3-mesitylpropan-2-ol hemihydrate

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The title compound, 2C16H27NOH2O, crystallizes in the monoclinic P21/c space group with two independent molecules (A and B) in the asymmetric unit. In the crystal, molecules A and B are linked through the water molecules by intermolecular O—H⋅⋅⋅O and O—H⋅⋅⋅N hydrogen bonds, producing chains along the b-axis direction. These chains are linked with neighboring chains parallel to the (103) plane via C—H⋅⋅⋅π interactions, generating ribbons along the b-axis direction. The stability of the molecular packaging is ensured by van der Waals interactions between the ribbons. According to the Hirshfeld surface study, H⋅⋅⋅H interactions are the most significant contributors to the crystal packing (80.3% for molecule A and 84.8% for molecule B).

1. Chemical context

Amine group-containing compounds are of great interest in the fields of organic synthesis, catalysis, material science and medicinal chemistry (Zubkov et al., 2018; Shikhaliev et al., 2019; Viswanathan et al., 2019; Gurbanov et al., 2020). In particular, the β-amino alcohol moiety is the predominant structural motif in a series of natural and synthetic biologically active molecules (Lee & Kang, 2004). Amino alcohol derivatives are currently being studied for their antimicrobial, antifungal, antioxidant, cytotoxic, enzyme inhibitory and other important biological activities, which have been well documented in recent works (Baker et al., 2021; Estolano-Cobián et al., 2020; Tafelska-Kaczmarek et al., 2020).

In this study, in the framework of ongoing structural studies (Safavora et al., 2019; Aliyeva et al., 2011; Mamedov et al., 2022), we report the crystal structure and Hirshfeld surface analysis of the title compound, 1-(tert-butylamino)-3-mesitylpropan-2-ol hemihydrate.
2. Structural commentary

The title compound (Fig. 1) contains the two independent molecules (molecule A containing atom N1 and molecule B containing N2) in the asymmetric unit. As shown in Fig. 2 (r.m.s. deviation = 0.006 Å), while the 1,2,3,5-tetramethylbenzene parts of molecules A and B are overlapped, their 2-(tert-butylamino)ethan-1-ol moieties do not overlap, but rather are oriented in opposite directions. Atoms C2 in molecule A and C18 in molecule B have opposite chiralities. The chirality about the C2 atom is R and that about C18, S. The values of the geometric parameters of molecules A and B are normal and compatible with those of the related compounds mentioned in the Database survey section.

3. Supramolecular features and Hirshfeld surface analysis

In the crystal, molecules A and B are linked through the water molecules by intermolecular O—H⋯O and O—H⋯N hydrogen bonds (Table 1; Figs. 3 and 4), forming chains along the b-axis direction. These chains are linked by C—H⋯π interactions with neighboring chains parallel to the (103) plane, forming ribbons along the b-axis direction (Table 1; Figs. 5 and 6). The stability of the molecular packing is ensured by van der Waals interactions between the ribbons.

Hirshfeld surfaces were generated for both independent molecules using Crystal Explorer 17 (Turner et al., 2017). The $d_{	ext{norm}}$ mappings for molecules A and B were performed in the

![Figure 1](image1.png)

**Figure 1**

View of the two independent molecules, A and B, in the asymmetric unit of the title compound, with displacement ellipsoids for the non-hydrogen atoms drawn at the 30% probability level. For clarity, the minor components of disorder in molecule B are omitted.

![Figure 2](image2.png)

**Figure 2**

Overlay image of the two independent molecules (A and B) in the asymmetric unit of the title compound. Both the major and minor components of disorder in molecule B are shown. Color code: carbon (gray), hydrogen (white), nitrogen (blue) and oxygen (red).

![Figure 3](image3.png)

**Figure 3**

A view of the intermolecular O—H⋯O and O—H⋯N interactions along the a axis in the crystal structure of the title compound. For clarity, H atoms not involved in hydrogen bonding and the minor disorder components in molecule B are omitted.

### Table 1

| Hydrogen-bond geometry (Å, °) | O1—H1O⋯O3 | D—H | H⋯D | D⋯A | A—D⋯H | $d_{	ext{norm}}$ |
|-----------------------------|-----------|-----|-----|-----|-------|---------------|
| O1—H1O⋯O3                  | 0.91 (2)  | 1.82 (2) | 2.725 (5) | 173 (2) |
| O1—H1O⋯O3'                 | 0.91 (2)  | 1.82 (2) | 2.697 (6) | 161 (2) |
| O2—H2O⋯N1                  | 0.91 (2)  | 1.83 (2) | 2.7273 (13) | 168.0 (19) |
| O3—H3C⋯O2'                 | 0.95 (2)  | 1.83 (2) | 2.753 (3) | 162 (2) |
| O3—H3D⋯N2                  | 0.98 (3)  | 1.87 (3) | 2.827 (3) | 164 (2) |
| O3—H3D⋯N2'                 | 1.07 (3)  | 1.87 (3) | 2.875 (3) | 155 (2) |
| C11—H11B⋯Cg2               | 0.98      | 2.90 | 3.7613 (17) | 147 |

Symmetry codes: (i) $x, y-1, z$; (ii) $-x+2, y-1, -z+1$.
ranges 0.6784 to 1.2952 a.u. and 0.6765 to 1.3828 a.u., respectively. The O—H · · · O and O—H · · · N interactions are indicated by red areas on the Hirshfeld surfaces (Fig. 7a,b for A and Fig. 7c,d for B).

Fingerprint plots (Fig. 8) reveal that while H · · · H interactions (80.3% for molecule A and 84.8% for molecule B) make the largest contributions to surface contacts (Tables 1 and 2), C · · · H/H · · · C contacts (13.0% for molecule A and
Table 2
Summary of short interatomic contacts (Å) in the title compound.

| Contact         | Distance | Symmetry operation |
|-----------------|----------|--------------------|
| O2···H3C        | 1.83     | x, 1 + y, z        |
| H2O···N1        | 1.83     | x, y, z            |
| N2···H3D        | 1.87     | x, y, z            |
| H26C···H15B     | 2.58     | 1 − x, 2 − y, 1 − z|
| *H31D···H17B    | 2.34     | x, −1 + y, z       |
| *H32E···H30E    | 2.50     | 1 − x, 1/2 + y, ½ − z|
| H24···H3B       | 2.39     | −1 + x, y, z       |
| H26B···H15C     | 2.58     | 1 − x, 1 − y, 1 − z|
| *H30C···C10     | 3.00     | x, −1 + y, z       |
| *H31B···H6      | 2.44     | 1 − x, −1/2 + y, ½ − z|
| *H32D···H11C    | 2.48     | 1 − x, 1/2 + y, ½ − z|
| H1O···*O3      | 1.82     | x, y, z            |
| H1O···H1B       | 2.46     | x, −1 + y, z       |
| C9···H11B       | 2.84     | 2 − x, ½ + y, ½ − z|
| H16C···*O3     | 2.89     | x, 1 + y, z        |

The prefix * denotes atoms of the disordered parts of the molecules.

6. Refinement
Crystal data, data collection and structure refinement details are summarized in Table 3. Carbon-bound H atoms were placed in calculated positions [C—H = 0.95 to 1.00 Å; Uiso(H) = 1.2 or 1.5Ueq(C)] and were included in the refinement in the riding-model approximation. The hydroxy and amino H atoms were located in a difference Fourier map, and were freely refined [O1—H1O = 0.91 (2) Å, O2—H2O = 0.91 (2) Å, N1—H1N = 0.922 (16) Å and N2—H2N = 0.922 (18) Å]. In molecule B, the methyl groups of the 2-methylpropane moiety are disordered over two sets of sites with an occupancy ratio of 0.65 (3):0.35 (3). The two H atoms of the water molecule were found in a difference-Fourier map and freely refined [O3—H3C = 0.95 (2) Å, O3—H3D = 0.98 (3) Å, O3’—H3C = 0.92 (2) Å and O3’—H3D =
Table 3
Experimental details.

| Crystal data         | 2C16H27NO·H2O |
|----------------------|---------------|
| M, g·mol⁻¹           | 516.79        |
| Crystal system, space group | Monoclinic, P2₁/c |
| Temperature (K)      | 100           |
| a, b, c (Å)          | 13.06508 (16), 5.81242 (6), 4.17384 (5) |
| β (°)                | 93.3315 (11)  |
| V (Å³)               | 3164.25 (6)   |
| Z                    | 4             |
| Radiation type       | Cu Ka         |
| μ (mm⁻¹)             | 0.53          |
| Crystal size (mm)    | 0.36 × 0.12 × 0.06 |

Data collection
Diffractometer
XtaLAB Synergy, Dualflex, HyPix
Absorption correction
Multi-scan (CrysAlis PRO; Rigaku OD, 2021)

| Tmin, Tmax         | 0.805, 0.941 |
| No. of measured, independent and observed [I > 2σ(I)] reflections | 40427, 6866, 6251 |
| R₁(ref)            | 0.041        |
| (sinθ/λ)max (Å⁻¹) | 0.638        |

Refinement
R[F² > 2σ(F²)], wR(F²), S 0.044, 0.114, 1.09
No. of reflections 6866
No. of parameters 411
No. of restraints 21
H-atom treatment H atoms treated by a mixture of independent and constrained refinement
Δρmax, Δρmin (e Å⁻³) 0.19, −0.20

Computer programs: CrysAlis PRO (Rigaku OD, 2021), SHEXLX (Sheldrick, 2015a), SHELXI (Sheldrick, 2015b), ORTEP-3 for Windows (Farrugia, 2012) and PLATON (Spek, 2020).

1.07 (3) Å. The anisotropic displacement parameters of the O3 and O3' atoms of the disordered water molecule were restrained to be equal (SIMU). SADI and DFIX commands were used for the treatment of the disordered methyl groups of the 2-methylpropano moiety of molecule B.

Acknowledgements
Authors’ contributions are as follows. Conceptualization, ANK and IGM; methodology, ANK and IGM; investigation, ANK, MA and TAT; writing (original draft), MA and ANK; writing (review and editing of the manuscript), MA and ANK; visualization, MA, ANK and IGM; funding acquisition, VNK, RMR and ANK; resources, AAA, VNK and RMR; supervision, ANK and MA.

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Crystal structure and Hirshfeld surface analysis of 1-(tert-butylamino)-3-mesitylpropan-2-ol hemihydrate

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Computing details

Data collection: *CrysAlis PRO* (Rigaku OD, 2021); cell refinement: *CrysAlis PRO* (Rigaku OD, 2021); data reduction: *CrysAlis PRO* (Rigaku OD, 2021); program(s) used to solve structure: *SHELXT* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015b); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *PLATON* (Spek, 2020).

1-(tert-Butylamino)-3-(2,4,6-trimethylphenyl)propan-2-ol hemihydrate

Crystal data

$2C_{16}H_{27}NO·H_2O$

$M_r = 516.79$

Monoclinic, *P*2$_1$/c

$a = 13.06508$ (16) Å

$b = 5.81242$ (6) Å

$c = 41.7384$ (5) Å

$\beta = 93.3315$ (11)$^\circ$

$V = 3164.25$ (6) Å$^3$

$Z = 4$

$F(000) = 1144$

$D_x = 1.085$ Mg m$^{-3}$

Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å

Cell parameters from 22446 reflections

$\theta = 2.2$–79.1$^\circ$

$\mu = 0.53$ mm$^{-1}$

$T = 100$ K

Plate, colourless

0.36 × 0.12 × 0.06 mm

Data collection

*XtaLAB Synergy, Dualflex, HyPix*

diffractometer

Radiation source: micro-focus sealed X-ray tube

$\varphi$ and $\omega$ scans

Absorption correction: multi-scan

(*CrysAlisPro; Rigaku OD, 2021*)

$T_{\text{min}} = 0.805$, $T_{\text{max}} = 0.941$

40427 measured reflections

6866 independent reflections

6251 reflections with $I > 2\sigma(I)$

$R_{	ext{int}} = 0.041$

$\theta_{\text{max}} = 79.7^\circ$, $\theta_{\text{min}} = 3.4^\circ$

$h = -16$→16

$k = -6$→7

$l = -53$→52

Refinement

Refinement on $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.044$

$wR(F^2) = 0.114$

$S = 1.09$

6866 reflections

411 parameters

21 restraints

Primary atom site location: difference Fourier map

Secondary atom site location: difference Fourier map

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

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supporting information

\[ w = \frac{1}{\sqrt{\sigma^2(F_o^2) + (0.0467P^2 + 0.9632P)}} \]
\[ \text{where } P = (F_o^2 + 2F_c^2)/3 \]
\[ (\Delta/\sigma)_{\text{max}} = 0.001 \]
\[ \Delta \rho_{\text{max}} = 0.19 \text{ e Å}^{-3} \]
\[ \Delta \rho_{\text{min}} = -0.20 \text{ e Å}^{-3} \]

**Special details**

**Experimental.** CrysAlisPro 1.171.41.117a (Rigaku OD, 2021) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

**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.

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

|     | x       | y       | z       | Uiso/ Ueq | Occ. (<1) |
|-----|---------|---------|---------|-----------|-----------|
| O1  | 0.83664 (7) | 0.63481 (14) | 0.38179 (2) | 0.02938 (18) |
| H1O | 0.7755 (17)  | 0.562 (4) | 0.3786 (5) | 0.068 (6)*  |
| N1  | 0.75396 (7)  | 0.93228 (16) | 0.42788 (2) | 0.02387 (19) |
| H1N | 0.7600 (11)  | 0.774 (3) | 0.4290 (3) | 0.031 (4)*  |
| C1  | 0.82855 (8)  | 1.01229 (19) | 0.40524 (3) | 0.0256 (2)  |
| H1A | 0.8989  | 0.9969 | 0.4152 | 0.031*     |
| H1B | 0.8163  | 1.1769 | 0.4003 | 0.031*     |
| C2  | 0.81900 (8)  | 0.87226 (18) | 0.37442 (3) | 0.0252 (2)  |
| H2  | 0.7481  | 0.8905 | 0.3644 | 0.030*     |
| C3  | 0.89639 (9)  | 0.9587 (2) | 0.35095 (3) | 0.0293 (2)  |
| H3A | 0.8870  | 1.1266 | 0.3481 | 0.035*     |
| H3B | 0.9665  | 0.9334 | 0.3606 | 0.035*     |
| C4  | 0.88794 (9)  | 0.8449 (2) | 0.31835 (3) | 0.0291 (2)  |
| C5  | 0.81775 (9)  | 0.9285 (2) | 0.29442 (3) | 0.0325 (3)  |
| C6  | 0.81180 (9)  | 0.8239 (2) | 0.26423 (3) | 0.0360 (3)  |
| H6  | 0.7642  | 0.8817 | 0.2481 | 0.043*     |
| C7  | 0.87307 (10) | 0.6384 (2) | 0.25699 (3) | 0.0363 (3)  |
| C8  | 0.94234 (10) | 0.5581 (2) | 0.28094 (3) | 0.0359 (3)  |
| H8  | 0.9851  | 0.4314 | 0.2764 | 0.043*     |
| C9  | 0.95115 (9)  | 0.6570 (2) | 0.31138 (3) | 0.0322 (3)  |
| C10 | 0.74982 (11) | 1.1329 (2) | 0.30008 (3) | 0.0393 (3)  |
| H10A| 0.7097  | 1.1034 | 0.3188 | 0.059*     |
| H10B| 0.7033  | 1.1580 | 0.2811 | 0.059*     |
| H10C| 0.7924  | 1.2700 | 0.3040 | 0.059*     |
| C11 | 0.86603 (12) | 0.5272 (3) | 0.22430 (3) | 0.0462 (3)  |
| H11A| 0.8362  | 0.3732 | 0.2259 | 0.069*     |
| H11B| 0.9348  | 0.5150 | 0.2162 | 0.069*     |
| H11C| 0.8225  | 0.6210 | 0.2095 | 0.069*     |
| C12 | 1.03108 (10) | 0.5649 (3) | 0.33569 (3) | 0.0400 (3)  |
| H12A| 1.0620  | 0.4256 | 0.3272 | 0.060*     |
| H12B| 0.9987  | 0.5279 | 0.3557 | 0.060*     |
| H12C| 1.0843  | 0.6814 | 0.3400 | 0.060*     |
| C13 | 0.76617 (8)  | 1.02608 (19) | 0.46103 (3) | 0.0247 (2)  |
| C14 | 0.86787 (9)  | 0.9562 (2) | 0.47850 (3) | 0.0336 (3)  |
| Atom  | x      | y      | z      | U(eq)  |
|-------|--------|--------|--------|--------|
| H14A  | 0.8726 | 0.7880 | 0.4794 | 0.050* |
| H14B  | 0.8710 | 1.0182 | 0.5004 | 0.050* |
| H14C  | 0.9250 | 1.0176 | 0.4669 | 0.050* |
| C15   | 0.67782 (9) | 0.9273 (2) | 0.47916 (3) | 0.0288 (2) |
| H15A  | 0.6125 | 0.9847 | 0.4695 | 0.043* |
| H15B  | 0.6853 | 0.9748 | 0.5017 | 0.043* |
| H15C  | 0.6790 | 0.7589 | 0.4779 | 0.043* |
| C16   | 0.75688 (10) | 1.2882 (2) | 0.45967 (3) | 0.0306 (2) |
| H16A  | 0.8151 | 1.3522 | 0.4488 | 0.046* |
| H16B  | 0.7568 | 1.3497 | 0.4815 | 0.046* |
| H16C  | 0.6928 | 1.3306 | 0.4478 | 0.046* |
| O2    | 0.57184 (6) | 1.06462 (16) | 0.39828 (2) | 0.0352 (2) |
| H2O   | 0.6278 (15) | 1.004 (3) | 0.4093 (5) | 0.062 (5)* |
| N2    | 0.50806 (8) | 0.78162 (18) | 0.34494 (2) | 0.0306 (2) |
| H2N   | 0.5598 (13) | 0.887 (3) | 0.3424 (4) | 0.047 (4)* |
| C17   | 0.43655 (8) | 0.8875 (2) | 0.36627 (3) | 0.0283 (2) |
| H17A  | 0.3746 | 0.7901 | 0.3671 | 0.034* |
| H17B  | 0.4151 | 1.0399 | 0.3577 | 0.034* |
| C18   | 0.48579 (8) | 0.91565 (19) | 0.39983 (3) | 0.0264 (2) |
| H18   | 0.5096 | 0.7620 | 0.4081 | 0.032* |
| C19   | 0.41002 (9) | 1.01744 (19) | 0.42282 (3) | 0.0271 (2) |
| H19A  | 0.4465 | 1.0414 | 0.4440 | 0.032* |
| H19B  | 0.3876 | 1.1702 | 0.4146 | 0.032* |
| C20   | 0.31558 (8) | 0.87209 (19) | 0.42747 (2) | 0.0244 (2) |
| C21   | 0.32295 (8) | 0.67813 (19) | 0.44783 (2) | 0.0246 (2) |
| C22   | 0.23623 (9) | 0.5454 (2) | 0.45241 (3) | 0.0269 (2) |
| H22   | 0.2424 | 0.4139 | 0.4659 | 0.032* |
| C23   | 0.14096 (9) | 0.5996 (2) | 0.43785 (3) | 0.0286 (2) |
| C24   | 0.13486 (8) | 0.7904 (2) | 0.41774 (3) | 0.0287 (2) |
| H24   | 0.0704 | 0.8298 | 0.4075 | 0.034* |
| C25   | 0.21998 (9) | 0.92618 (19) | 0.41200 (3) | 0.0264 (2) |
| C26   | 0.42242 (9) | 0.6101 (2) | 0.46563 (3) | 0.0288 (2) |
| H26A  | 0.4727 | 0.5667 | 0.4502 | 0.043* |
| H26B  | 0.4103 | 0.4793 | 0.4797 | 0.043* |
| H26C  | 0.4488 | 0.7403 | 0.4785 | 0.043* |
| C27   | 0.04768 (9) | 0.4554 (2) | 0.44353 (3) | 0.0369 (3) |
| H27A  | 0.0240 | 0.4888 | 0.4649 | 0.055* |
| H27B  | 0.0654 | 0.2920 | 0.4422 | 0.055* |
| H27C  | -0.0070 | 0.4917 | 0.4272 | 0.055* |
| C28   | 0.20503 (9) | 1.1238 (2) | 0.38851 (3) | 0.0317 (2) |
| H28A  | 0.2394 | 1.2613 | 0.3974 | 0.047* |
| H28B  | 0.1316 | 1.1549 | 0.3847 | 0.047* |
| H28C  | 0.2344 | 1.0828 | 0.3682 | 0.047* |
| C29   | 0.46433 (9) | 0.7090 (2) | 0.31290 (3) | 0.0363 (3) |
| C30   | 0.5508 (4) | 0.5955 (11) | 0.29559 (17) | 0.074 (2) |
| H30A  | 0.6091 | 0.7013 | 0.2952 | 0.111* |
| H30B  | 0.5266 | 0.5583 | 0.2735 | 0.111* |
| H30C  | 0.5722 | 0.4540 | 0.3069 | 0.111* |
| Atomic displacement parameters (Å²) | \(U_{11}^1\) | \(U_{22}^2\) | \(U_{33}^3\) | \(U_{12} \) | \(U_{13} \) | \(U_{23} \) |
|-----------------------------------|--------------|--------------|--------------|-------------|-------------|-------------|
| \(O1\)                           | 0.0295 (4)   | 0.0246 (4)   | 0.0344 (4)   | 0.0009 (3)  | 0.0046 (3)  | 0.0014 (3)  |
| \(N1\)                           | 0.0240 (4)   | 0.0230 (4)   | 0.0249 (4)   | −0.0032 (3) | 0.0039 (3)  | −0.0005 (3) |
| \(C1\)                           | 0.0234 (5)   | 0.0268 (5)   | 0.0270 (5)   | −0.0030 (4) | 0.0039 (4)  | 0.0010 (4)  |
| \(C2\)                           | 0.0232 (5)   | 0.0250 (5)   | 0.0277 (5)   | 0.0005 (4)  | 0.0038 (4)  | 0.0019 (4)  |
| \(C3\)                           | 0.0270 (5)   | 0.0315 (6)   | 0.0300 (6)   | −0.0026 (4) | 0.0063 (4)  | 0.0010 (5)  |
| \(C4\)                           | 0.0259 (5)   | 0.0331 (6)   | 0.0290 (5)   | −0.0024 (4) | 0.0086 (4)  | 0.0021 (4)  |
| \(C5\)                           | 0.0287 (6)   | 0.0386 (6)   | 0.0312 (6)   | 0.0008 (5)  | 0.0095 (4)  | 0.0042 (5)  |
| \(C6\)                           | 0.0307 (6)   | 0.0487 (7)   | 0.0291 (6)   | 0.0007 (5)  | 0.0058 (5)  | 0.0030 (5)  |
| \(C7\)                           | 0.0313 (6)   | 0.0458 (7)   | 0.0327 (6)   | −0.0048 (5) | 0.0099 (5)  | −0.0035 (5) |
| \(C8\)                           | 0.0301 (6)   | 0.0398 (7)   | 0.0388 (6)   | 0.0019 (5)  | 0.0105 (5)  | −0.0044 (5) |
| \(C9\)                           | 0.0257 (5)   | 0.0375 (6)   | 0.0342 (6)   | 0.0003 (5)  | 0.0081 (4)  | 0.0004 (5)  |
| \(C10\)                          | 0.0409 (7)   | 0.0437 (7)   | 0.0341 (6)   | 0.0098 (6)  | 0.0088 (5)  | 0.0077 (5)  |
| \(C11\)                          | 0.0438 (7)   | 0.0593 (9)   | 0.0363 (7)   | −0.0035 (7) | 0.0082 (6)  | −0.0105 (6) |
| \(C12\)                          | 0.0322 (6)   | 0.0471 (7)   | 0.0408 (7)   | 0.0089 (5)  | 0.0036 (5)  | −0.0019 (6) |
| \(C13\)                          | 0.0251 (5)   | 0.0251 (5)   | 0.0240 (5)   | −0.0023 (4) | 0.0009 (4)  | 0.0000 (4)  |
| \(C14\)                          | 0.0292 (6)   | 0.0405 (6)   | 0.0305 (6)   | −0.0014 (5) | −0.0024 (4) | 0.0030 (5)  |
| \(C15\)                          | 0.0311 (6)   | 0.0297 (5)   | 0.0260 (5)   | −0.0032 (4) | 0.0046 (4)  | 0.0002 (4)  |
| \(C16\)                          | 0.0377 (6)   | 0.0258 (5)   | 0.0285 (5)   | −0.0035 (5) | 0.0028 (5)  | −0.0026 (4) |
| \(O2\)                           | 0.0249 (4)   | 0.0390 (5)   | 0.0412 (5)   | −0.0096 (3) | −0.0023 (3) | 0.0157 (4)  |
| \(N2\)                           | 0.0274 (5)   | 0.0342 (5)   | 0.0307 (5)   | 0.0032 (4)  | 0.0048 (4)  | 0.0018 (4)  |
supporting information

Geometric parameters (Å, °)

| Bond      | Distance (Å) | Angle (°)  |
|-----------|--------------|------------|
| O1—C2    | 1.4298 (13)  |            |
| O1—H1O   | 0.91 (2)     |            |
| N1—C1    | 1.4718 (13)  |            |
| N1—C13   | 1.4872 (14)  |            |
| N1—H1N   | 0.922 (16)   |            |
| C1—C2    | 1.5211 (15)  |            |
| C1—H1A   | 0.9900       |            |
| C1—H1B   | 0.9900       |            |
| C2—C3    | 1.5330 (15)  |            |
| C2—H2    | 1.0000       |            |
| C3—C4    | 1.5117 (16)  |            |
| C3—H3A   | 0.9900       |            |
| C3—H3B   | 0.9900       |            |
| C4—C5    | 1.4030 (17)  |            |
| C4—C9    | 1.4096 (17)  |            |
| C5—C6    | 1.3972 (18)  |            |
| C5—C10   | 1.5097 (18)  |            |
| C6—C7    | 1.3863 (19)  |            |
| C6—H6    | 0.9500       |            |
| C7—C8    | 1.3896 (19)  |            |
| C7—C11   | 1.5081 (18)  |            |
| C8—C9    | 1.3930 (18)  |            |
| C8—H8    | 0.9500       |            |
| C9—C12   | 1.5112 (18)  |            |

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C10—H10A 0.9800  C28—H28C 0.9800
C10—H10B 0.9800  C29—C32' 1.525 (3)
C10—H10C 0.9800  C29—C30 1.526 (2)
C11—H11A 0.9800  C29—C31' 1.527 (3)
C11—H11B 0.9800  C29—C32 1.527 (2)
C11—H11C 0.9800  C29—C30' 1.528 (3)
C12—H12A 0.9800  C29—C31 1.529 (2)
C12—H12B 0.9800  C30—H30A 0.9800
C12—H12C 0.9800  C30—H30B 0.9800
C13—C15 1.5284 (15)  C30—H30C 0.9800
C13—C16 1.5289 (16)  C31—H31A 0.9800
C13—C14 1.5328 (15)  C31—H31B 0.9800
C14—H14A 0.9800  C31—H31C 0.9800
C14—H14B 0.9800  C32—H32A 0.9800
C14—H14C 0.9800  C32—H32B 0.9800
C15—H15A 0.9800  C32—H32C 0.9800
C15—H15B 0.9800  C30'—H30D 0.9800
C15—H15C 0.9800  C30'—H30E 0.9800
C16—H16A 0.9800  C30'—H30F 0.9800
C16—H16B 0.9800  C31'—H31D 0.9800
C16—H16C 0.9800  C31'—H31E 0.9800
O2—C18 1.4235 (13)  C31'—H31F 0.9800
O2—H2O 0.91 (2)  C32'—H32D 0.9800
N2—C17 1.4631 (15)  C32'—H32E 0.9800
N2—C29 1.4846 (16)  C32'—H32F 0.9800
O1—C2—C1 109.29 (9)  O3—H3C 0.95 (2)
C1—N1—C13 116.25 (8)  C17—C18—H18 109.1
C1—N1—H1N 106.8 (9)  C20—C19—C18 115.06 (9)
C13—N1—H1N 106.8 (9)  C19—C18—H18 109.1
N1—C1—C2 110.42 (9)  C20—C19—H19A 108.5
N1—C1—H1A 109.6  C18—C19—H19A 108.5
C2—C1—H1A 109.6  C20—C19—H19B 108.5
N1—C1—H1B 109.6  C18—C19—H19B 108.5
C2—C1—H1B 109.6  H19A—C19—H19B 107.5
H1A—C1—H1B 108.1  C25—C20—C21 118.82 (10)
O1—C2—C1 110.48 (9)  C25—C20—C19 121.57 (10)
C1—C2—C3 109.88 (9)  C21—C20—C19 119.61 (10)
C2—C1—C2 109.1  C22—C21—C20 119.82 (10)
C2—C2—H2 109.1  C22—C21—C26 118.08 (10)
C1—C2—H2 109.1  C20—C21—C26 122.09 (10)
C3—C2—H2 109.1  C23—C22—C21 121.98 (11)
C4—C3—C2 115.54 (9)  C23—C22—H22 119.0
C4—C3—H3A 108.6  C21—C22—H22 119.0
| Bond                        | Angle (°) | Bond                        | Angle (°) |
|-----------------------------|-----------|-----------------------------|-----------|
| C2—C3—H3A                  | 108.6     | C22—C23—C24                | 117.61    |
| C4—C3—H3B                  | 108.6     | C22—C23—C27                | 121.08    |
| C2—C3—H3B                  | 108.6     | C24—C23—C27                | 121.30    |
| H3A—C3—H3B                 | 107.6     | C23—C24—C25                | 122.45    |
| C5—C4—C9                   | 119.21    | C23—C24—H24                | 118.8     |
| C5—C4—C3                   | 119.98    | C25—C24—H24                | 118.8     |
| C9—C4—C3                   | 120.79    | C24—C25—C20                | 119.30    |
| C6—C5—C4                   | 119.40    | C24—C25—C28                | 117.76    |
| C6—C5—C10                  | 118.77    | C20—C25—C28                | 122.93    |
| C4—C5—C10                  | 121.81    | C21—C26—H26A               | 109.5     |
| C7—C6—C5                   | 122.16    | C21—C26—H26B               | 109.5     |
| C7—C6—H6                   | 118.9     | H26A—C26—H26B              | 109.5     |
| C5—C6—H6                   | 118.9     | C21—C26—H26C               | 109.5     |
| C6—C7—C8                   | 117.70    | H26A—C26—H26C              | 109.5     |
| C6—C7—C11                  | 121.66    | H26B—C26—H26C              | 109.5     |
| C8—C7—C11                  | 120.64    | C23—C27—H27A               | 109.5     |
| C7—C8—C9                   | 122.21    | C23—C27—H27B               | 109.5     |
| C7—C8—H8                   | 118.9     | H27A—C27—H27B              | 109.5     |
| C9—C8—H8                   | 118.9     | C23—C27—H27C               | 109.5     |
| C8—C9—C4                   | 119.31    | H27A—C27—H27C              | 109.5     |
| C8—C9—C12                  | 118.91    | H27B—C27—H27C              | 109.5     |
| C4—C9—C12                  | 121.74    | C25—C28—H28A               | 109.5     |
| C5—C10—H10A                | 109.5     | C25—C28—H28B               | 109.5     |
| C5—C10—H10B                | 109.5     | H28A—C28—H28B              | 109.5     |
| H10A—C10—H10B              | 109.5     | C25—C28—H28C               | 109.5     |
| C5—C10—H10C                | 109.5     | H28A—C28—H28C              | 109.5     |
| H10A—C10—H10C              | 109.5     | H28B—C28—H28C              | 109.5     |
| H10B—C10—H10C              | 109.5     | N2—C29—C32′                | 116.0 (6) |
| C7—C11—H11A                | 109.5     | N2—C29—C30                 | 107.0 (3) |
| C7—C11—H11B                | 109.5     | N2—C29—C31′                | 106.0 (6) |
| H11A—C11—H11B              | 109.5     | C32′—C29—C31′              | 109.9 (3) |
| C7—C11—H11C                | 109.5     | N2—C29—C32                 | 110.3 (3) |
| H11A—C11—H11C              | 109.5     | C30—C29—C32                | 109.9 (2) |
| H11B—C11—H11C              | 109.5     | N2—C29—C30′                | 105.3 (5) |
| C9—C12—H12A                | 109.5     | C32′—C29—C30′              | 109.8 (3) |
| C9—C12—H12B                | 109.5     | C31′—C29—C30′              | 109.7 (3) |
| H12A—C12—H12B              | 109.5     | N2—C29—C31                 | 110.2 (3) |
| C9—C12—H12C                | 109.5     | C30—C29—C31                | 109.6 (2) |
| H12A—C12—H12C              | 109.5     | C32—C29—C31                | 109.7 (2) |
| H12B—C12—H12C              | 109.5     | C29—C30—H30A               | 109.5     |
| N1—C13—C15                 | 106.23 (8)| C29—C30—H30B               | 109.5     |
| N1—C13—C16                 | 109.07 (9)| H30A—C30—H30B              | 109.5     |
| C15—C13—C16                | 109.40 (9)| C29—C30—H30C               | 109.5     |
| N1—C13—C14                 | 112.86 (9)| H30A—C30—H30C              | 109.5     |
| C15—C13—C14                | 108.89 (9)| H30B—C30—H30C              | 109.5     |
| C16—C13—C14                | 110.27 (10)| C29—C31—H31A              | 109.5     |
| C13—C14—H14A               | 109.5     | C29—C31—H31B               | 109.5     |
| C13—C14—H14B               | 109.5     | H31A—C31—H31B              | 109.5     |
| Bond/Angle | Value (°) | Bond/Angle | Value (°) | Bond/Angle | Value (°) |
|-----------|----------|-----------|----------|-----------|----------|
| H14A—C14—H14B | 109.5 | C29—C31—H31C | 109.5 | H14A—C14—H14C | 109.5 |
| C13—C14—H14C | 109.5 | H31A—C31—H31C | 109.5 | H14B—C14—H14C | 109.5 |
| H14C—C14—H14C | 109.5 | C29—C32—H32A | 109.5 | C13—C15—H15A | 109.5 |
| C13—C15—H15A | 109.5 | C29—C32—H32B | 109.5 | C13—C15—H15B | 109.5 |
| H15A—C15—H15B | 109.5 | C29—C32—H32C | 109.5 | H15A—C15—H15C | 109.5 |
| C13—C15—H15C | 109.5 | H32A—C32—H32B | 109.5 | H15B—C15—H15C | 109.5 |
| C13—C16—H16A | 109.5 | C29—C32—H32E | 109.5 | C13—C16—H16B | 109.5 |
| C13—C16—H16B | 109.5 | H30D—C30′—H30E | 109.5 | C16A—C16—H16C | 109.5 |
| C16A—C16—H16C | 109.5 | H30E—C30′—H30F | 109.5 | H16B—C16—H16C | 109.5 |
| C18—O2—H2O | 110.6 (13) | C29—C31′—H31E | 109.5 | C18—O2—H2O | 110.6 (13) |
| C17—N2—C29 | 116.22 (9) | C31D—C31′—H31E | 109.5 | C17—N2—C29 | 116.22 (9) |
| C17—N2—H2N | 106.8 (11) | C29—C31′—H31F | 109.5 | C17—N2—H2N | 106.8 (11) |
| C29—N2—H2N | 109.4 (11) | H31D—C31′—H31F | 109.5 | C29—N2—H2N | 109.4 (11) |
| N2—C17—C18 | 110.86 (9) | H31E—C31′—H31F | 109.5 | N2—C17—C18 | 110.86 (9) |
| N2—C17—H17A | 109.5 | C31′—H31E | 109.5 | N2—C17—H17A | 109.5 |
| C18—C17—H17A | 109.5 | C31—H31F | 109.5 | C18—C17—H17A | 109.5 |
| N2—C17—H17B | 109.5 | C31′—H31F | 109.5 | N2—C17—H17B | 109.5 |
| C18—C17—H17B | 109.5 | H32D—C32′—H32E | 109.5 | C18—C17—H17B | 109.5 |
| H17A—C17—H17B | 108.1 | C32—C32′—H32F | 109.5 | H17A—C17—H17B | 108.1 |
| O2—C18—C17 | 108.52 (9) | H32E—C32′—H32F | 109.5 | O2—C18—C17 | 108.52 (9) |
| O2—C18—C19 | 109.47 (9) | H3C—O3—H3D | 100.9 (19) | O2—C18—C19 | 109.47 (9) |
| C17—C18—C19 | 111.38 (9) | H3C—O3′—H3D | 97 (2) | C17—C18—C19 | 111.38 (9) |

| Bond/Angle | Value (°) | Bond/Angle | Value (°) | Bond/Angle | Value (°) |
|-----------|----------|-----------|----------|-----------|----------|
| C13—N1—C1—C2 | 169.98 (9) | N2—C17—C18—C19 | −177.64 (9) | C13—C16—H16C | 109.5 |
| N1—C1—C2—O1 | −59.13 (11) | O2—C18—C19—C20 | −178.38 (9) | C13—C16—H16C | 109.5 |
| N1—C1—C2—C3 | 179.49 (9) | C17—C18—C19—C20 | 61.61 (12) | C13—C16—H16C | 109.5 |
| O1—C2—C3—C4 | 64.12 (12) | C18—C19—C20—C25 | −102.29 (12) | C13—C16—H16C | 109.5 |
| C1—C2—C3—C4 | −175.21 (10) | C18—C19—C20—C21 | 77.93 (13) | C13—C16—H16C | 109.5 |
| C2—C3—C4—C5 | 85.11 (13) | C25—C20—C21—C22 | −0.39 (16) | C13—C16—H16C | 109.5 |
| C2—C3—C4—C9 | −95.98 (13) | C19—C20—C21—C22 | 179.39 (10) | C13—C16—H16C | 109.5 |
| C9—C4—C5—C6 | 0.10 (17) | C25—C20—C21—C26 | −179.24 (10) | C13—C16—H16C | 109.5 |
| C3—C4—C5—C6 | 179.03 (11) | C19—C20—C21—C26 | 0.55 (16) | C13—C16—H16C | 109.5 |
| C9—C4—C5—C10 | −178.41 (11) | C20—C21—C22—C23 | −1.02 (17) | C13—C16—H16C | 109.5 |
| C3—C4—C5—C10 | 0.52 (17) | C26—C21—C22—C23 | 177.87 (10) | C13—C16—H16C | 109.5 |
| C4—C5—C6—C7 | 0.16 (17) | C21—C22—C23—C24 | 1.29 (17) | C13—C16—H16C | 109.5 |
| C10—C5—C6—C7 | 178.71 (12) | C21—C22—C23—C27 | −179.10 (11) | C13—C16—H16C | 109.5 |
| C5—C6—C7—C8 | −0.30 (19) | C22—C23—C24—C25 | −0.17 (17) | C13—C16—H16C | 109.5 |
| C5—C6—C7—C11 | −179.92 (12) | C27—C23—C24—C25 | −179.78 (11) | C13—C16—H16C | 109.5 |
| C6—C7—C8—C9 | 0.20 (19) | C23—C24—C25—C20 | −1.20 (17) | C13—C16—H16C | 109.5 |
| C11—C7—C8—C9 | 179.81 (12) | C23—C24—C25—C28 | 177.42 (11) | C13—C16—H16C | 109.5 |
| C7—C8—C9—C4 | 0.05 (19) | C21—C20—C25—C24 | 1.46 (16) | C13—C16—H16C | 109.5 |
C7—C8—C9—C12  -177.80 (12)  C19—C20—C25—C24  -178.32 (10)
C5—C4—C9—C8  -0.20 (17)  C21—C20—C25—C28  -177.09 (10)
C3—C4—C9—C8  -179.12 (11)  C19—C20—C25—C28  3.13 (16)
C5—C4—C9—C12  177.59 (11)  C17—N2—C29—C32'  52.0 (7)
C3—C4—C9—C12  -1.33 (17)  C17—N2—C29—C30  -177.7 (3)
C5—C4—C9—C12  177.59 (11)  C17—N2—C29—C30  3.13 (16)
C1—N1—C13—C15  177.41 (9)  C17—N2—C29—C30  -177.7 (3)
C1—N1—C13—C16  59.59 (12)  C17—N2—C29—C30  62.7 (3)
C1—N1—C13—C15  -63.33 (12)  C17—N2—C29—C30  173.6 (6)
C29—N2—C17—C18  170.22 (10)  C17—N2—C29—C31  -58.6 (3)
N2—C17—C18—O2  61.79 (12)

Hydrogen-bond geometry (Å, °)

$C_g$ is the centroid of the benzene ring (C4–C9) of molecule A.

| D—H···A   | D—H | H···A | D···A | D—H···A |
|-----------|-----|-------|-------|----------|
| O1—H1O···O3   | 0.91 (2) | 1.82 (2) | 2.725 (5) | 173 (2) |
| O1—H1O···O3'  | 0.91 (2) | 1.82 (2) | 2.697 (6) | 161 (2) |
| O2—H2O···N1   | 0.91 (2) | 1.83 (2) | 2.7273 (13) | 168.0 (19) |
| O3—H3C···O2i  | 0.95 (2) | 1.83 (2) | 2.753 (3) | 162 (2) |
| O3'—H3C'···O2i| 0.92 (2) | 1.83 (2) | 2.685 (4) | 153 (2) |
| O3—H3D···N2   | 0.98 (3) | 1.87 (3) | 2.827 (3) | 164 (2) |
| O3'—H3D'···N2 | 1.07 (3) | 1.87 (3) | 2.875 (5) | 155 (2) |
| C11—H11B···$C_g$ii | 0.98 | 2.90 | 3.7613 (17) | 147 |

Symmetry codes: (i) x, y−1, z; (ii) −x+2, y−1/2, −z+1/2.