Crystal structure and Hirshfeld surface analysis of \((\pm)-N'-(2-hydroxy-3-methoxybenzylidene)-2-(4-isobutylphenyl)propionohydrazide\)

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The title molecule, C\textsubscript{21}H\textsubscript{26}N\textsubscript{2}O\textsubscript{3}, adopts a V-shaped conformation and is chiral at the C atom with methyl group attached at the common cut of the edges of the V-conformation and crystallizes as a racemate. It also contains an intramolecular O—H\cdots N hydrogen bond. In the crystal, N—H\cdots O hydrogen bonds form chains of molecules extending along the c-axis direction, together with normal van der Waals contacts. The roles of the various intermolecular interactions were clarified by Hirshfeld surface analysis, which reveals that the most important contributions to the crystal packing are from H\cdots H (62.6\%), C\cdots H/ H\cdots C (15.8\%) and O\cdots H/H\cdots O (15.3\%) contacts.

1. Chemical context
Non-steroidal anti-inflammatory drugs (NSAIDs) are commonly used as analgesics and antipyretics to manage pain and inflammation in people with chronic pain, osteoarthritis, rheumatoid arthritis, postoperative surgical conditions, and menstrual cramps (Manzano \textit{et al}., 2018; Gupta & Bah, 2016; Budoff, 1979). Azo-methine structure-based ibuprofen core compounds in particular have been used as anti-viral and antibacterial agents (El Bakri \textit{et al}., 2022). Based on such significant activity, we herein report the crystal structure of a member of this family, namely \((\pm)-N'-(2-hydroxy-3-methoxybenzylidene)-2-(4-isobutylphenyl)propionohydrazide.\n
2. Structural commentary
In the solid state, the molecule adopts a wide, V-shaped conformation (Fig. 1) with a dihedral angle of 1.08 (11)° between the mean plane of the C1–C6 ring and the chain
Table 1
Hydrogen-bond geometry (Å, °).

| Contact        | D—H   | D—A   | H···A | D···A | D—H···A |
|----------------|-------|-------|-------|-------|---------|
| O1···HO1···N1  | 0.85  | 1.83  | 1.38  | 2.5914(13) | 149(2) |
| N2···HN2···O1' | 0.90  | 2.40  | 1.34  | 3.2470(14) | 157(1) |
| N2···HN2···O2' | 0.90  | 2.18  | 1.28  | 2.8745(14) | 133(1) |

Symmetry code: (i) x, −y + 1, −z − 1.

Table 2
Summary of short interatomic contacts (Å) in the title compound.

| Contact        | Distance | Symmetry operation |
|----------------|----------|--------------------|
| HO1···H7C      | 2.49     | 1 − x, − 1/2 + y, 1/2 − z |
| O2···HN2      | 2.18     | 1/2 − x, y, 1/2 + z |
| H13···H7C     | 2.47     | 1 − x, 2/3 − y, 1/3 − z |
| H11B···C2     | 2.95     | 1 − x, 1 − y, 1 − z |
| C6···H19A     | 2.90     | 1 + x, 1/2 − y, 1/2 + z |
| H6···C13      | 2.98     | 1 − x, 1/2 + y, 1/2 − z |
| H7A···H20D    | 2.26     | 1 + x, y, 1 + z |
| H11A···H20F   | 2.05     | −x, −1/2 + y, 1/2 − z |
| H20C···H14    | 2.58     | −x, 2 − y, −z |
| H21D···H21A   | 2.02     | −x, 1 − y, −z |

Figure 1
The title molecule with labeling scheme and 30% probability level ellipsoids. The intramolecular O—H···N hydrogen bond is depicted by a dashed line. Only the major component of the disorder is shown.

Figure 2
A portion of one chain viewed along the b-axis with the O—H···N and N···H···O hydrogen bonds depicted by dashed lines and non-interacting hydrogen atoms omitted for clarity.

provided by normal van der Waals interactions between chains.

Hirshfeld surfaces and their related two-dimensional fingerprint plots were generated using CrystalExplorer17.5 (Turner et al., 2017) to visually represent the intermolecular interactions in the crystal structure of the title compound. The Hirshfeld surface plotted over dnorm in the range −0.3801 to +1.4738 a.u. is shown in Fig. 3. The interactions shown in Tables 1 and 2 are important in the molecular packing of the title compound.

The overall two-dimensional fingerprint plot is illustrated in Fig. 4a, and those delineated into the major contacts: H···H (62.6%; Fig. 4b), C···H/H···C (15.8%; Fig. 4c), O···H/H···O and (15.3%; Fig. 4d). The other contacts are negligible with individual contributions of less than 2.2% [N···H/H···N (2.2%), N···C/C···N (2.1%), C···C (1.3%) and N···C/C···N (0.7%)].

4. Database survey
Six related compounds were found in a search of the Cambridge Structural Database (CSD, version 5.42, update of

Figure 3
(a) Front view and (b) back view of the three-dimensional Hirshfeld surface of the title compound plotted over dnorm in the range −0.3801 to +1.4738 a.u. The red, white and blue regions visible on the dnorm surfaces indicate contacts with distances shorter, longer and equal to the van der Waals separations, respectively. The red spots highlight the interatomic contacts, including the O—H···N and N···H···O hydrogen bonds.
September 2021; Groom et al., 2016, viz. N'-benzylidene-2-((5-([4-chlorophenoxymethyl]-4-phenyl-4H-1,2,4-triazol-3-yl)sulfanyl)acetohydrazide hemihydrate [CSD refcode ULARIK (I); Mague et al., 2016], N'-[(3-cyanophenyl)methyldiene]-N-methyl-2-(thiophen-2-yl)acetohydrazide [ECOWEB (II); Cardoso et al., 2017], N'-[(4-methoxyphenyl)methyldiene]-N-methyl-2-(thiophen-2-yl)acetohydrazide [ECOWIF (III); Cardoso et al., 2017], N'-(1H)-1-(3-methyl-5-oxo-1-phenyl-1,5-dihydro-4H-pyrrozol-4-ylidene)ethyl]-2-[4-(methylphenyl)sulfanyl]acetohydrazide [GEMQIB (IV); Mohamed et al., 2017], (E)-N'-(4-fluorobenzylidene)-2-(3-methylphenyl)acetohydrazide [MEWMUY (V); Praveen et al., 2013] and N'-[4-(dimethylamino)benzylidene]-2-[4-(methylphenoxycarbonyl)acetohydrazide [ZIYSOR (VI); Usha et al., 2014].

In (I), three independent molecules in the asymmetric unit and two water molecules of crystallization are observed. The three unique organic molecules differ in the conformations of the substituents on the pyrazole ring. In the crystal, extensive O—H—O, O—H—N, N—H—O and C—H—O hydrogen bonding generates a three-dimensional network and C—H—O interactions are also observed. Compounds (II) and (III) crystallize with two molecules in the asymmetric unit, with generally similar conformations that approximate to L-shapes. The packing for (II) features short C—H···O interactions arising from the C—H adjacent to the cyanide group and C—H···N (c = cyanide) links arising from the methine groups to generate [110] double chains. Weak C—H···π interactions interlink the chains into a three-dimensional network. The packing for (III) features numerous C—H···O and C—H···π interactions arising from different donor groups to generate a three-dimensional network. In (IV), the molecular conformation is influenced by intramolecular N—H···O and C—H···O hydrogen bonds. In the crystal, N—H···O hydrogen bonds plus C—H···π and π···π stacking interactions lead to the formation of chains extending in the a-axis direction. The chains are linked by complementary pairs of C—H···π interactions. Compound (V) has four independent molecules in the asymmetric unit. In the crystal, N—H···O hydrogen bonds involving the hydrazide and acetyl groups, which form R2(18) ring motifs, link the molecules into dimers, which form columns along the [010] plane. In the crystal of (VI), the molecules are linked by C—H···O and N—H···O hydrogen bonds, as well as weak C—H···π contacts, forming a three-dimensional supramolecular architecture.

5. Synthesis and crystallization

The title compound was synthesized by mixing 1.101 g (5 mmol) of ibuprofen hydrazide in 15 mL of chloroform with 0.76 g (5 mmol) of 2-hydroxy-3-methoxybenzaldehyde in 15 mL of methanol. A few drops of acetic acid were added to the reaction mixture as catalyst and the mixture was refluxed at 333 K for 1 h. The reaction progress was monitored by TLC until completion. The crude product as a pale-yellow precipitate was filtered off, washed, recrystallized from ethanol and dried under vacuum over anhydrous CaCl2 under vacuum. M.p. 444.15 K; 87% yield.

The product was characterized by different spectroscopic analyses. Empirical formula, C21H26N2O3 (354.33 g mol⁻¹); IR (cm⁻¹): 3280 (NH), 1704 (C=O), 1612 (C=O), and 1248 (C—H vibrations). 1H NMR (400 MHz, CDCl3) ppm δ = 0.83–0.86 (d, J = 6.6 Hz, 6H), 1.37–1.43 (d, J = 7.0 Hz, 3H), 1.45–1.84 (m, 1H), 2.37–2.52 (d, J = 7.1 Hz, 2H), 3.65–3.70 (q, J = 7.0 Hz, 3H), 3.80–3.82 (s, 3H), 6.81–7.30 (m, 7H), 8.41 (s, 1H), 10.82 (s, 1H), 11.73 (s, 1H). 13C NMR (75 MHz, CDCl3) δ = 18.88, 19.10, 22.64, 30.10, 39.55, 39.97, 40.38, 44.70, 56.28, 113.19, 114.12, 118.34, 119.68, 121.12, 127.68, 129.46, 139.72, 140.14, 146.31, 148.34, 170.12.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. H atoms attached to carbon were placed in calculated positions (C—H = 0.95–1.00 Å) and were included as riding contributions with isotropic displacement parameters 1.2–1.5 times those of the attached atoms. Those attached to nitrogen and to oxygen were placed in locations derived from a difference map and refined freely with DFIX 0.91 0.01 and DFIX 0.84 0.01 instructions, respectively. The atoms of the propane group are disordered over two sets of sites with an occupancy ratio of 0.929 (3):0.071 (3).
Table 3
Experimental details.

| Crystal data | Chemical formula | C_{21}H_{26}N_{2}O_{3} |
|--------------|------------------|-------------------------|
| M_r          | 354.44           |                         |
| Crystal system, space group | Monoclinic, P2_1/c |
| Temperature (K) | 125              |
| a, b, c (Å)   | 14.5241 (7), 10.0718 (5), 13.2710 (7) |
| β (°)         | 97.042 (2)       |
| V (Å^3)       | 1926.69 (17)     |
| Z             | 4                |
| Radiation type | Cu Kα            |
| μ (mm⁻¹)      | 0.66             |
| Crystal size (mm) | 0.20 x 0.20 x 0.03 |

| Diffractometer | Bruker D8 VENTURE PHOTON |
|----------------|--------------------------|
| Absorption correction | Multi-scan (SADABS; Krause et al., 2015) |
| T_min, T_max     | 0.90, 0.98               |
| No. of measured, independent and observed | 38584, 3758, 3400 |
| R_{int}         | 0.035                     |
| (sin θ/λ)_{max}  | 0.618                     |

| Refinement | \(R(F^2 > 2σ(F^2))\), wR(F^2), S |
|------------|----------------------------------|
| No. of reflections | 3758          |
| No. of parameters | 259            |
| No. of restraints | 8              |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement |
| Δρ_{max}, Δρ_{min} (e Å⁻³) | 0.40, −0.21 |

Computer programs: APEX3 and SAINT (Bruker, 2016), SHELXT (Sheldrick, 2015a), SHELXL2018/1 (Sheldrick, 2015b), ORTEP-3 for Windows (Farrugia, 2012) and PLATON (Spek, 2020).

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Author contributions to this paper are as follows: synthesis and organic chemistry parts preparation, MAH, MRA; EAAT; conceptualization and study guide, LHAR, SKM; financial support, EAA; paper preparation and Hirshfeld study, MA, SKM.

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Crystal structure and Hirshfeld surface analysis of (±)-N'-[(2-hydroxy-3-methoxybenzylidene)-2-(4-isobutylphenyl)propionohydrazide

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Computing details

Data collection: APEX3 (Bruker, 2016); cell refinement: SAINT (Bruker, 2016); data reduction: SAINT (Bruker, 2016); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: SHELXL2018/1 (Sheldrick, 2015b); molecular graphics: ORTEP-3 for Windows (Farrugia, 2012); software used to prepare material for publication: PLATON (Spek, 2020).

(±)-N'-(2-Hydroxy-3-methoxybenzylidene)-2-(4-isobutylphenyl)propionohydrazide

Crystal data

C_{21}H_{26}N_{2}O_{3}  
Mr = 354.44  
Monoclinic, P2_{1}/c  
a = 14.5241 (7) Å  
b = 10.0718 (5) Å  
c = 13.2710 (7) Å  
β = 97.042 (2)°  
V = 1926.69 (17) Å³  
Z = 4  

F(000) = 760  
D_{x} = 1.222 Mg m⁻³  
Cu Kα radiation, λ = 1.54178 Å  
Cell parameters from 9778 reflections  
θ = 5.5–72.4°  
µ = 0.66 mm⁻¹  
T = 125 K  
Plate, colourless  
0.20 × 0.20 × 0.03 mm

Data collection

Bruker D8 VENTURE PHOTON 3 CPAD  
diffractometer  
Radiation source: INCOATEC IμS micro-focus source  
Mirror monochromator  
Detector resolution: 7.3910 pixels mm⁻¹  
φ and ω scans  
(SADABS; Krause et al., 2015)

38584 measured reflections  
3758 independent reflections  
3400 reflections with I > 2σ(I)  
R_{int} = 0.035  
θ_{max} = 72.4°, θ_{min} = 5.5°  
h = −17→17  
k = −12→12  
l = −14→16

Refinement

Refinement on F²  
Least-squares matrix: full  
R[F² > 2σ(F²)] = 0.041  
wR(F²) = 0.109  
S = 1.06  
3758 reflections  
259 parameters  
8 restraints

Hydrogen site location: mixed  
H atoms treated by a mixture of independent and constrained refinement  
w = 1/[σ(F²) + (0.0517P)² + 0.694P]  
where P = (F² + 2Fc²)/3  
(Δρ)_{max} < 0.001  
Δρ_{max} = 0.40 e Å⁻³  
Δρ_{min} = −0.21 e Å⁻³
**Special details**

**Experimental.** The diffraction data were obtained from 15 sets of frames, each of width 0.5° in $\omega$ or $\varphi$, collected with scan parameters determined by the "strategy" routine in APEX4. The scan time was 10 sec/frame.

**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 ($\AA^2$)**

| Atomic symbol | x        | y        | z        | $U_{iso}^*$/U$_{eq}$ | Occ. (<1) |
|---------------|----------|----------|----------|----------------------|-----------|
| O1            | 0.48117  (6) | 0.83125 (10) | 0.62950 (7) | 0.0328 (2)          |           |
| HO1           | 0.4586 (13) | 0.7973 (19)  | 0.5735 (10) | 0.061 (6)*           |           |
| O2            | 0.57158 (7)  | 0.95029 (10) | 0.78175 (7) | 0.0378 (2)           |           |
| O3            | 0.30167 (6)  | 0.64479 (10) | 0.44100 (7) | 0.0353 (2)           |           |
| N1            | 0.46923 (7)  | 0.75956 (10) | 0.44081 (8) | 0.0276 (2)           |           |
| N2            | 0.42448 (7)  | 0.69530 (11) | 0.35782 (8) | 0.0291 (2)           |           |
| N2            | 0.4561 (10)  | 0.6800 (17)  | 0.3046 (10) | 0.041 (4)*           |           |
| C1            | 0.59869 (9)  | 0.88043 (12) | 0.51948 (9) | 0.0272 (3)           |           |
| C2            | 0.56349 (8)  | 0.88651 (12) | 0.61296 (9) | 0.0259 (3)           |           |
| C3            | 0.61408 (9)  | 0.95151 (12) | 0.69520 (9) | 0.0276 (3)           |           |
| C4            | 0.69830 (9)  | 1.01023 (13)| 0.68500 (10)| 0.0310 (3)           |           |
| C5            | 0.732144 | 1.054394 | 0.741015 | 0.037*               |           |
| C6            | 0.73341 (9)  | 1.00426 (15)| 0.59170 (11)| 0.0364 (3)           |           |
| C7            | 0.791294 | 1.044688 | 0.584278 | 0.044*               |           |
| C8            | 0.68465 (9)  | 0.94019 (14)| 0.51054 (10)| 0.0341 (3)           |           |
| H5            | 0.709452 | 0.936307 | 0.447606 | 0.041*               |           |
| C9            | 0.624321 | 1.113185 | 0.849937 | 0.051*               |           |
| C10           | 0.54787 (9)  | 0.81416 (13)| 0.43243 (9) | 0.0289 (3)           |           |
| C11           | 0.572764 | 0.811269 | 0.369529 | 0.035*               |           |
| H8            | 0.34188 (9)  | 0.63537 (13)| 0.36611 (9) | 0.0277 (3)           |           |
| C12           | 0.30483 (9)  | 0.55529 (13)| 0.27208 (9) | 0.0293 (3)           |           |
| C13           | 0.352167 | 0.557419 | 0.223374 | 0.035*               |           |
| C14           | 0.29224 (12)| 0.41096 (14)| 0.30427 (12)| 0.0427 (4)           |           |
| H11A          | 0.250084 | 0.407883 | 0.356498 | 0.064*               |           |
| H11B          | 0.352538 | 0.373853 | 0.331631 | 0.064*               |           |
| H11C          | 0.266064 | 0.358829 | 0.245264 | 0.064*               |           |
| C15           | 0.21646 (8)  | 0.61920 (12)| 0.22138 (9) | 0.0268 (3)           |           |
| C16           | 0.21756 (9)  | 0.68811 (13)| 0.13073 (9) | 0.0287 (3)           |           |
| H13           | 0.273825 | 0.694597 | 0.101326 | 0.034*               |           |
| C14           | 0.13798 (10)| 0.74751 (13)| 0.08254 (10)| 0.0340 (3)           |           |
| H14           | 0.140562 | 0.794801 | 0.021011 | 0.041*               |           |
| C15           | 0.05427 (10)| 0.73865 (15)| 0.12330 (10)| 0.0377 (3)           |           |
| C16           | 0.05360 (10)| 0.66893 (17)| 0.21379 (11)| 0.0419 (4)           |           |
| H16           | −0.002864 | 0.660731 | 0.242608 | 0.050*               |           |
supporting information

### Atomic displacement parameters (Å²)

|     | U₁₁   | U₂₂   | U₃₃   | U₁₂   | U₁₃   | U₂₃   |
|-----|-------|-------|-------|-------|-------|-------|
| O1  | 0.0293 (5) | 0.0422 (5) | 0.0276 (5) | -0.0105 (4) | 0.0059 (4) | -0.0063 (4) |
| O2  | 0.0384 (5) | 0.0494 (6) | 0.0260 (5) | -0.0148 (4) | 0.0054 (4) | -0.0083 (4) |
| O3  | 0.0315 (5) | 0.0495 (6) | 0.0245 (5) | -0.0024 (4) | 0.0017 (4) | -0.0011 (4) |
| N1  | 0.0287 (5) | 0.0295 (5) | 0.0238 (5) | 0.0024 (4) | -0.0005 (4) | -0.0023 (4) |
| N2  | 0.0290 (5) | 0.0352 (6) | 0.0225 (5) | 0.0011 (4) | 0.0012 (4) | -0.0047 (4) |
| C1  | 0.0279 (6) | 0.0265 (6) | 0.0270 (6) | 0.0017 (5) | 0.0025 (5) | 0.0029 (5) |
| C2  | 0.0246 (6) | 0.0252 (6) | 0.0278 (6) | -0.0002 (5) | 0.0025 (5) | 0.0027 (5) |
| C3  | 0.0291 (6) | 0.0278 (6) | 0.0257 (6) | 0.0003 (5) | 0.0024 (5) | 0.0020 (5) |
| C4  | 0.0291 (6) | 0.0307 (6) | 0.0315 (6) | -0.0022 (5) | -0.0028 (5) | 0.0013 (5) |
| C5  | 0.0287 (6) | 0.0412 (7) | 0.0395 (7) | -0.0078 (6) | 0.0052 (5) | 0.0017 (6) |
| C6  | 0.0330 (7) | 0.0397 (7) | 0.0306 (7) | -0.0043 (6) | 0.0080 (5) | 0.0022 (5) |
| C7  | 0.0349 (7) | 0.0394 (7) | 0.0275 (6) | -0.0025 (6) | -0.0008 (5) | -0.0065 (5) |
| C8  | 0.0308 (6) | 0.0306 (6) | 0.0255 (6) | 0.0013 (5) | 0.0039 (5) | 0.0016 (5) |
| C9  | 0.0286 (6) | 0.0309 (6) | 0.0226 (6) | 0.0050 (5) | -0.0005 (5) | 0.0015 (5) |
| C10 | 0.0290 (6) | 0.0327 (7) | 0.0255 (6) | 0.0021 (5) | 0.0000 (5) | -0.0024 (5) |
| C11 | 0.0522 (9) | 0.0326 (7) | 0.0414 (8) | 0.0032 (6) | -0.0021 (7) | -0.0002 (6) |
| C12 | 0.0277 (6) | 0.0281 (6) | 0.0235 (6) | -0.0009 (5) | -0.0009 (5) | -0.0044 (5) |
| C13 | 0.0287 (6) | 0.0303 (6) | 0.0270 (6) | -0.0027 (5) | 0.0029 (5) | -0.0034 (5) |
| C14 | 0.0414 (7) | 0.0330 (7) | 0.0265 (6) | 0.0032 (6) | -0.0002 (5) | 0.0003 (5) |
### Geometric parameters (Å, °)

|        |        |        |        |        |        |        |        |        |        |
|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|
| O1—C2  | 1.3609 (15) | C13—C14 | 1.3863 (19) |        |        |        |        |        |        |
| O1—HO1 | 0.847 (9)   | C13—H13 | 0.9500  |        |        |        |        |        |        |
| O2—C3  | 1.3690 (15) | C14—C15 | 1.393 (2) |        |        |        |        |        |        |
| O2—C7  | 1.4265 (15) | C14—H14 | 0.9500  |        |        |        |        |        |        |
| O3—C9  | 1.2167 (16) | C15—C16 | 1.392 (2) |        |        |        |        |        |        |
| N1—C8  | 1.2847 (17) | C15—C18 | 1.5146 (19) |        |        |        |        |        |        |
| N1—N2  | 1.3706 (14) | C16—C17 | 1.381 (2) |        |        |        |        |        |        |
| N2—C9  | 1.3594 (17) | C16—H16 | 0.9500  |        |        |        |        |        |        |
| C1—C2  | 1.3999 (17) | C17—H17 | 0.9500  |        |        |        |        |        |        |
| C1—C6  | 1.4042 (18) | C18—C19 | 1.453 (8) |        |        |        |        |        |        |
| C1—C8  | 1.4545 (17) | C18—H18A | 0.9900 |        |        |        |        |        |        |
| C2—C3  | 1.4011 (17) | C18—H18B | 0.9900 |        |        |        |        |        |        |
| C3—C4  | 1.3802 (18) | C19—C21 | 1.505 (3) |        |        |        |        |        |        |
| C4—C5  | 1.397 (2)   | C19—C20 | 1.529 (2) |        |        |        |        |        |        |
| C4—H4  | 0.9500     | C19—H19 | 1.0000  |        |        |        |        |        |        |
| C5—C6  | 1.3751 (19) | C20—H20A | 0.9800 |        |        |        |        |        |        |
| C5—H5  | 0.9500     | C20—H20B | 0.9800 |        |        |        |        |        |        |
| C6—H6  | 0.9500     | C20—H20C | 0.9800 |        |        |        |        |        |        |
| C7—H7A | 0.9800     | C21—H21A | 0.9800 |        |        |        |        |        |        |
| C7—H7B | 0.9800     | C21—H21B | 0.9800 |        |        |        |        |        |        |
| C7—H7C | 0.9800     | C21—H21C | 0.9800 |        |        |        |        |        |        |
| C8—H8  | 0.9500     | C19A—C21A | 1.50 (3) |        |        |        |        |        |        |
| C9—C10 | 1.5272 (17) | C19A—C20A | 1.50 (3) |        |        |        |        |        |        |
| C10—C12 | 1.5174 (17) | C19A—H19A | 1.0000 |        |        |        |        |        |        |
| C10—C11 | 1.5324 (19) | C20A—H20D | 0.9800 |        |        |        |        |        |        |
| C10—H10 | 1.0000    | C20A—H20E | 0.9800 |        |        |        |        |        |        |
| C11—H11A | 0.9800   | C20A—H20F | 0.9800 |        |        |        |        |        |        |
| C11—H11B | 0.9800   | C21A—H21D | 0.9800 |        |        |        |        |        |        |
| C11—H11C | 0.9800   | C21A—H21E | 0.9800 |        |        |        |        |        |        |
| C12—C17 | 1.3896 (18) | C21A—H21F | 0.9800 |        |        |        |        |        |        |
| C12—C13 | 1.3907 (18) |        |        |        |        |        |        |        |        |
| C2—O1—HO1 | 106.1 (14) |        |        |        |        |        |        |        |        |
| C3—O2—C7 | 117.83 (10) | C13—C14—H14 | 119.6 |        |        |        |        |        |        |

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C8—N1—N2 118.14 (11)  C16—C15—C14 117.78 (12)
C9—N2—N1 118.74 (10)  C16—C15—C18 119.51 (14)
C9—N2—HN2 121.8 (11)  C14—C15—C18 122.71 (14)
N1—N2—HN2 118.3 (11)  C17—C16—C15 121.48 (13)
C2—C1—C6 118.76 (12)  C17—C16—H16 119.3
C2—C1—C8 121.27 (11)  C15—C16—H16 119.3
C6—C1—C8 119.97 (11)  C16—C17—C12 120.70 (13)
O1—C2—C1  123.27 (11)  C16—C17—H17 119.6
C2—C1—C6 118.76 (12)  C12—C17—H17 119.6
C2—C1—C8 121.27 (11)  C19—C18—C15 128.4 (7)
C6—C1—C8 119.97 (11)  C19—C18—C15 116.46 (13)
O1—C2—C3 116.91 (11)  C19—C18—H18A 108.2
O1—C2—C3 116.91 (11)  C15—C18—H18A 108.2
C3—C4—C5 119.52 (12)  C19—C18—H18B 108.2
C3—C4—C5 119.52 (12)  C15—C18—H18B 108.2
C5—C4—C5 120.2  H18A—C18—H18B 107.3
C5—C4—C5 120.2  H18A—C18—H18B 107.3
C6—C5—C4 120.36 (12)  C21—C19—C18 113.35 (14)
C6—C5—C4 120.36 (12)  C21—C19—C20 110.26 (15)
C6—C5—C4 120.36 (12)  C12—C19—C20 109.95 (14)
C6—C5—H5 119.8  C21—C19—H19 107.7
C6—C5—H5 119.8  C18—C19—C20 107.7
C6—C5—H5 119.8  C20—C19—C20 107.7
C4—C3—C2 120.68 (12)  C19—C21—C18 109.5
C4—C3—C2 120.68 (12)  C19—C21—C18 109.5
C3—C4—H4 120.2  C19—C21—H18A 109.5
C3—C4—H4 120.2  C19—C21—H18B 109.5
H4—C3—C4 120.2  C19—C21—H18B 109.5
H4—C3—C4 120.2  C19—C21—H18B 109.5
C5—C6—C1 120.86 (12)  C21—C19—C20 109.5
C5—C6—C1 120.86 (12)  C21—C19—H19 107.7
C5—C6—C1 120.86 (12)  C21—C19—H19 107.7
C5—C6—H6 119.6  C18—C19—H19 107.7
C5—C6—H6 119.6  C20—C19—H19 107.7
C1—C6—H6 119.6  C19—C20—C18 109.5
O2—C7—H7A 109.5  C19—C20—H20A 109.5
O2—C7—H7B 109.5  C19—C20—H20A 109.5
H7B—C7—H7A 109.5  C19—C20—H20A 109.5
H7A—C7—H7B 109.5  C19—C20—H20A 109.5
H7A—C7—H7B 109.5  C19—C20—H20A 109.5
N1—C8—C1 119.85 (11)  C19—C21—H21A 109.5
N1—C8—H8 120.1  C19—C21—H21A 109.5
C1—C8—H8 120.1  C19—C21—H21A 109.5
O3—C9—N2 123.24 (12)  C19—C21—H21A 109.5
O3—C9—N2 123.24 (12)  C19—C21—H21A 109.5
O3—C9—C10 123.52 (12)  C19—C21—H21A 109.5
O3—C9—C10 123.52 (12)  C19—C21—H21A 109.5
N2—C9—C10 113.23 (11)  C19—C21—H21B 109.5
C12—C10—H10 108.4  C19—C21—H21B 109.5
C12—C10—H10 108.4  C19—C21—H21B 109.5
C12—C10—H10 108.4  C19—C21—H21B 109.5
C12—C10—H10 108.4  C19—C21—H21B 109.5
C10—C11—H11A 109.5  C19—C21—H21B 109.5
C10—C11—H11A 109.5  C19—C21—H21B 109.5
H11A—C11—H11B 109.5  C19—C21—H21B 109.5
H11A—C11—H11B 109.5  C19—C21—H21B 109.5
C10—C11—H11C 109.5  C19—C21—H21B 109.5
C10—C11—H11C 109.5  C19—C21—H21B 109.5
H11B—C11—H11C 109.5  C19—C21—H21B 109.5
H11B—C11—H11C 109.5  C19—C21—H21B 109.5
C17—C12—C13 118.13 (12)  C19—C21—H21B 109.5
C17—C12—C13 118.13 (12)  C19—C21—H21B 109.5
C17—C12—C10 122.10 (11)  C19—C21—H21B 109.5
C13—C12—C10 119.77 (11)  H21D—C21A—H21E 109.5
C14—C13—C12 121.14 (12)  C19A—C21A—H21F 109.5
C14—C13—H13 119.4  H21D—C21A—H21F 109.5
C12—C13—H13 119.4  H21E—C21A—H21F 109.5
C13—C14—C15 120.75 (13)

C8—N1—N2—C9 179.02 (11)  O3—C9—C10—C11 −58.49 (16)
C6—C1—C2—O1 −179.68 (12)  N2—C9—C10—C11 −73.14 (15)
C8—C1—C2—O1 0.51 (19)  C11—C10—C12—C17 48.22 (17)
C6—C1—C2—C3 0.03 (18)  C9—C10—C12—C17 106.89 (13)
C8—C1—C2—C3 −179.78 (11)  H21D—C21A—H21F 109.5
C7—O2—C3—C4 2.85 (19)  C11—C10—C12—C13 −131.75 (13)
C7—O2—C3—C4 −179.78 (11)  C17—C12—C13—C14 −0.01 (19)
C8—C1—C2—C3 0.25 (18)  O1—C2—C3—O2 −179.97 (11)
C6—C1—C2—C3 −179.78 (11)  C12—C13—C14—C15 0.7 (2)
C8—C1—C2—C3 −179.78 (11)  C13—C14—C15—C16 −131.75 (13)
C7—O2—C3—C4 −179.78 (11)  C17—C12—C13—C14 −0.01 (19)
O1—C2—C3—O2 0.51 (19)  C10—C12—C13—C14 −179.00 (13)
C6—C1—C2—C3 −179.78 (11)  C12—C13—C14—C15 0.7 (2)
C8—C1—C2—C3 0.25 (18)  C13—C14—C15—C16 179.73 (13)
C7—O2—C3—C4 2.85 (19)  C14—C15—C16—C17 −1.3 (2)
C7—O2—C3—C4 −179.78 (11)  C13—C14—C15—C16 −179.73 (13)
C8—C1—C2—C3 0.25 (18)  O1—C2—C3—C4 −179.97 (11)
C6—C1—C2—C3 −179.78 (11)  C12—C13—C14—C15 0.7 (2)
C8—C1—C2—C3 −179.78 (11)  C13—C14—C15—C16 −131.75 (13)
C7—O2—C3—C4 2.85 (19)  C14—C15—C16—C17 179.73 (13)
C7—O2—C3—C4 −179.78 (11)  C13—C14—C15—C16 −179.73 (13)
C8—C1—C2—C3 0.25 (18)  O1—C2—C3—C4 −179.97 (11)
C6—C1—C2—C3 −179.78 (11)  C12—C13—C14—C15 0.7 (2)
C8—C1—C2—C3 −179.78 (11)  C13—C14—C15—C16 −131.75 (13)
C7—O2—C3—C4 2.85 (19)  C14—C15—C16—C17 179.73 (13)

Hydrogen-bond geometry (Å, °)

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|------|-------|-------|---------|
| O1—H01···N1 | 0.85 (1) | 1.83 (1) | 2.5914 (13) | 149 (2) |
| N2—H2···O1i | 0.90 (1) | 2.40 (1) | 3.2470 (14) | 157 (1) |
| N2—H2···O2i | 0.90 (1) | 2.18 (1) | 2.8745 (14) | 133 (1) |
| C10—H10···O1i | 1.00 | 2.63 | 3.5545 (16) | 155 |

Symmetry code: (i) x, −y+3/2, z−1/2.