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

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The title molecule, C21H26N2O3, 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···N hydrogen bond. In the crystal, N—H···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···H (62.6%), C···H/H···H (15.8%) and O···H/H···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 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 et al., 2022). Based on such significant activity, we herein report the crystal structure of a member of this family, namely (±)-N’-(2-hydroxy-3-methoxybenzylidene)-2-(4-isobutylphenyl)propionohydrazide.

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 Cl–C6 ring and the chain.
defined by C8, C9, N1 and N2. This is likely due to the intramolecular O1—H1···N1 hydrogen bond (Table 1 and Fig. 1). The dihedral angle between the latter chain and the mean plane of the C12–C17 ring is 59.34 (6)°. There is one stereogenic center in the racemic title compound and the chirality of the C10 atom is $S$ in the chosen asymmetric unit. All bond distances and angles appear as expected.

3. Supramolecular features and Hirshfeld surface analysis

In the crystal, N2—H2···O2 and weaker N2—H2···O1 hydrogen bonds (Table 1) form chains of molecules extending along the $c$-axis direction (Fig. 2). The molecular packing is 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 $d_{norm}$ 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%), C···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

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**Table 1**

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<th>Symmetry operation</th>
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<td>2.90 (1)</td>
<td>$1 - x, -rac{1}{2} + y, -z$</td>
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<tr>
<td>N2—H2···O1</td>
<td>2.95 (1)</td>
<td>$1 - x, -y, z$</td>
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<tr>
<td>N2—H2···O1'</td>
<td>2.90 (1)</td>
<td>$1 - x, -y, z + rac{1}{2}$</td>
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</table>

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<td>N2—H2···O1</td>
<td>2.95 (1)</td>
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<tr>
<td>N2—H2···O1'</td>
<td>2.90 (1)</td>
<td>$1 - x, -y, z + rac{1}{2}$</td>
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</table>

Symmetry code: (i) $x, -y + rac{1}{2}, z - rac{1}{2}$.
The substituents on the pyrazole ring. In the crystal, extensive
three unique organic molecules differ in the conformations of
and two water molecules of crystallization are observed. The
hydrazide [ZIYSOR (VI)] 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 R22(8) 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 1.101g
(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) and 1628, 1612 (C=N), and 1248 (C—
O). 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), 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 C21H26N2O3
Mr 354.44
Crystal system, space group Monoclinic, P21/c
Temperature (K) 125
a, b, c (Å) 14.5241 (7), 10.0718 (5), 13.2710 (7)
β (°) 97.042 (2)
V (Å³) 1926.69 (17)
Z 4
Radiation type Cu Kα
μ (mm⁻¹) 0.66
Crystal size (mm) 0.20 × 0.20 × 0.03

Data collection
Diffractometer Bruker D8 VENTURE PHOTON 3 CPAD
Absorption correction Multi-scan (SADABS; Krause et al., 2015)
 Tmin, Tmax 0.90, 0.98
No. of measured, independent and observed [I > 2σ(I)] reflections 38584, 3758, 3400
Rint 0.035
(sin θ/λ)max (Å⁻¹) 0.618

Refinement
R[F² > 2σ(F²)], wR(F²), S 0.041, 0.109, 1.06
No. of parameters 259
No. of restraints 8
H-atom treatment H atoms treated by a mixture of independent and constrained refinement
Δρmax, Δρmin (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).

Acknowledgements
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.

Funding information
The support of NSF–MRI grant No. 1228232 for the purchase of the diffractometer and Tulane University for support of the Tulane Crystallography Laboratory are gratefully acknowledged.

References
Bruker (2016). APEX3 and SAINT. Bruker AXS LLC, Madison, Wisconsin, USA.
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
C21H26N2O3
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 μS 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_min = 0.90, R_max = 0.98

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(F_c)^2 + (0.0517P)^2 + 0.694P]
where P = (F_c^2 + 2F_s^2)/3

(Δ/σ)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 (\( \text{Å}^2 \))

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Atomic displacement parameters (Å²)

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Symmetry code: (i) x, −y+3/2, z−1/2.