

Crystal structure, DFT and Hirshfeld surface analysis of *N*-acetyl-*t*-3-methyl-*r*-2,6-diphenylpiperidine

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Received 19 November 2021

Accepted 7 January 2022

Edited by J. Reibenspies, Texas A & M University, USA

Keywords: crystal structure; 2,6-substituted piperidine; hydrogen bonds; DFT.

CCDC reference: 2133146

Supporting information: this article has supporting information at journals.iucr.org/e

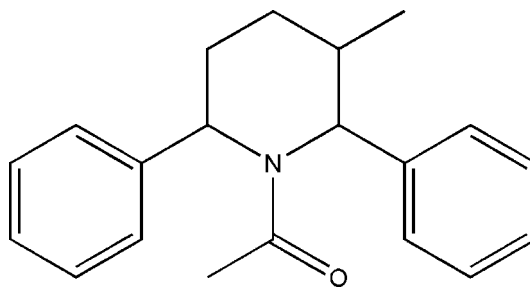
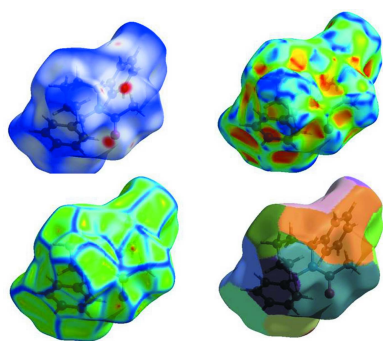
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In the title compound [systematic name: 1-(3-methyl-2,6-diphenylpiperidin-1-yl)ethanone], C₂₀H₂₃NO, the piperidine ring adopts a distorted boat conformation, while the phenyl rings subtend a dihedral angle 65.1 (2)°. In the crystal, molecules are linked by C–H···O hydrogen bonds into chains extending along the *b*-axis direction. The DFT/B3LYP/6–311 G(d,p) method was used to determine the HOMO–LUMO energy levels. A Hirshfeld surface analysis was conducted to verify the contributions of the different intermolecular interactions, indicating that the important contributions to the crystal packing are from H···H (73.2%), C···H (18.4%) and O···H (8.4%) interactions.

1. Chemical context

The structures of a wide array of heterocyclic derivatives have been analysed for their pharma-potentiality over the past three decades (Katritzky, 2010). Among these, derivatives of the six-membered heterocyclic base piperidine have proven to be successful pharmacophores. 2,6-Substituted piperidine derivatives have been found to be useful as tranquilizers and possess a wide range of biological activities such as anti-tumor (Vinaya *et al.*, 2009), antiviral, antimalarial, antibacterial and antifungal activities (Aridoss *et al.*, 2009; Mobio *et al.*, 1989). These have spurred considerable awareness of the synthetic arena based on their structure, reactivity, synthesis and biological properties. We report herein the crystal structure, Hirshfeld surface analysis and DFT computational calculations of the title compound.



2. Structural commentary

The methyl-substituted piperidine title compound crystallizes in the monoclinic space group *P*₂₁. A perspective view of the

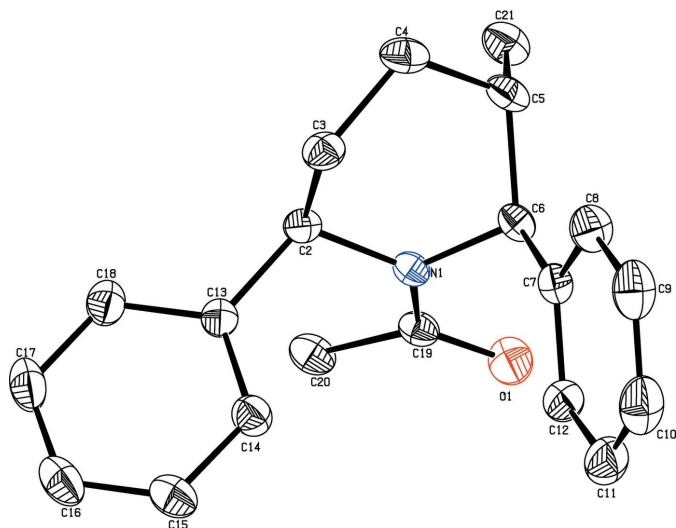


Figure 1
The molecular structure of the title compound, showing the atomic numbering and displacement ellipsoids drawn at the 30% probability level.

molecule is shown in Fig. 1. The bond lengths and angles are well within the expected limits (Roques *et al.*, 1981), and agree with values observed in related structures (Sekar *et al.*, 1990).

The piperidine ring adopts a distorted boat conformation with puckering parameters (Cremer & Pople, 1975) and asymmetry parameters (Nardelli, 1983): $q_2 = 0.720$ (2) Å, $q_3 = -0.004$ (3) Å, $\Phi(2) = 108.5$ (2)°, $\Delta C_s(C3)$ and $\Delta C_s(C6) = 14.5$ (2)°, and with maximum deviations of 0.406 (3) and 0.409 (2) Å, respectively, for atoms C3 and C6 from the best plane of the piperidine ring. The title molecule contains three chiral centres *viz.*, C2, C5 and C6. The absolute configuration of the chiral centres is assigned as C2 (*R*), C5 (*S*) and C6 (*S*). The parent molecule itself is chiral and the configuration cannot be changed during the substitution of acetyl group at the nitrogen.

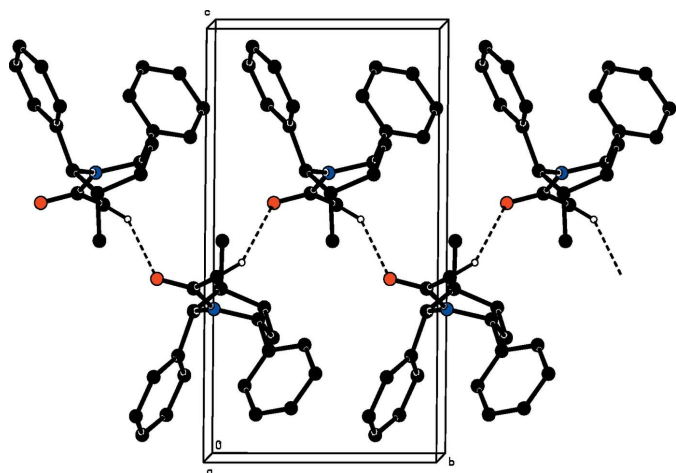


Figure 2
A partial view along the *b* axis of the crystal packing of the title compound, showing the formation of a molecular chain by C–H...O interactions (dotted lines).

The sum of the bond angles (358.2°) at atom N1 of the piperidine ring is in accordance with the sp^2 hybridization state (Beddoes *et al.*, 1986). The phenyl rings at the 2 and 6-positions of the piperidine ring occupy equatorial and axial orientations. The corresponding torsion angles are $C4-C3-C2-C13 = -178.8$ (2)° and $C4-C5-C6-C7 = -74.5$ (3)°.

The piperidine ring [N1/C2–C6] makes dihedral angles of 82.0 (1) and 58.4 (1)°, respectively, with the C13–C18 and C7–C12 phenyl rings, and confirms the fact that the moieties are in axial and equatorial orientations. It is to be noted that there is a possibility of resonance between atoms N1, C19 and O1 as a result of the delocalization of the hetero π electrons of the carbonyl group, which is also confirmed by the torsion angles $C2-N1-C19-O1 = 177.7$ (2)° and $C6-N1-C19-O1 = 13.0$ (3)°.

The methyl group substituted at the 5-position of the piperidine ring is axially oriented, as confirmed by the torsion angles $N1-C6-C5-C21 = -68.0$ (3)° and $C3-C4-C5-C21 = 112.4$ (3)°, whereas the methyl group substituted at C19

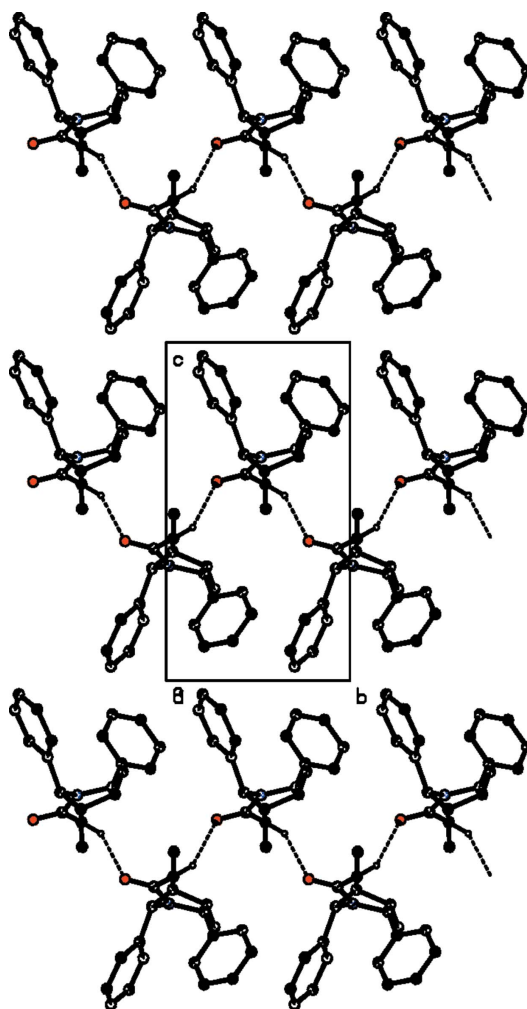


Figure 3
The overall crystal packing of the title compound, viewed along the *b* axis. Hydrogen bonds are shown as dashed lines, and only the H atoms involved in hydrogen bonding have been included.

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C20-H20B\cdots O1^i$	0.96	2.44	3.292 (3)	148

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

is oriented equatorially with torsion angle $C20-C19-N1-C6 = -166.3 (2)^\circ$ and $C20-C19-N1-C2 = -1.7 (3)^\circ$.

3. Supramolecular features

The crystal packing features $C-H\cdots O$ interactions (Table 1). Atom C20 of the molecule at (x, y, z) donates a proton to atom O1 of the molecule at $(-x + 1, y + \frac{1}{2}, -z + 1)$, forming a $C4$ zigzag chain (Bernstein *et al.*, 1995) running along the b -axis direction as shown in Fig. 2. The overall packing is shown in Fig. 3.

4. Density functional theory (DFT) study

The optimized molecular structure and frontier molecular orbitals (FMOs) (Figs. 4 and 5, respectively) were calculated using the DFT/B3LYP/6-311G(d,p) basis set implemented in the GAUSSIAN09 program package (Frisch *et al.*, 2009). The highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital (LUMO) are called frontier molecular orbitals (FMOs) as they lie at the outermost boundaries of the electrons of the molecule. The electron distribution (ED) of the HOMO-1, HOMO, LUMO and LUMO+1 energy levels and the energy values are shown in Fig. 5. The positive and negative phases are represented in green and red, respectively.

The HOMO of the title molecule is localized on one aromatic ring and the $C=O$ group, while the LUMO is located over the whole molecule with the exception of the CH_3 group and some carbon and hydrogen atoms in the piperidine ring. Thus the HOMO/LUMO implies an ED transfer to the $C=O$ group from the ring. The energy band gap ($\Delta E =$

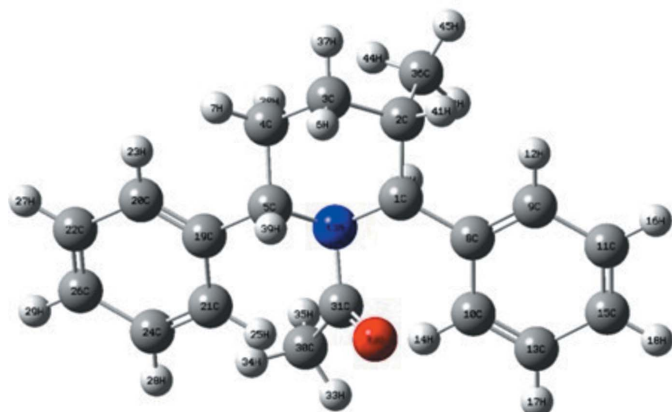


Figure 4
The optimized molecular structure of the title compound.

Table 2
Physico-chemical properties.

Parameter	Value
E_{HOMO} (eV)	-5.212
E_{LUMO} (eV)	-2.047
$E_{HOMO} - E_{LUMO}$ energy gap (eV)	3.165
E_{HOMO-1} (eV)	-5.851
E_{LUMO+1} (eV)	-2.248
$E_{HOMO-1} - E_{LUMO+1}$ energy gap (eV)	3.603
Ionization potential I (eV)	5.212
Electron affinity (A)	2.047
Electrophilicity Index (ω)	4.163
Chemical Potential (μ)	3.629
Electro negativity (χ)	-3.630
Hardness (η)	1.583
Softness (σ)	0.316

$E_{HOMO} - E_{LUMO}$) of the molecule is 3.165 eV and the calculated frontier molecular orbital energies, E_{HOMO} and E_{LUMO} , are -5.212 and -2.047 eV, respectively. The title compound has a small frontier orbital gap, hence the molecule has high chemical reactivity and low kinetic stability. The electron affinity (A) and ionization potential (I) of the molecule were calculated using the DFT/B3LYP/6-311++G(d,p) basis set. The values of the hardness (η), softness (σ), electronegativity (χ) and electrophilicity index (ω) for the title compound are given in Table 2.

5. Hirshfeld surface analysis

In order to visualize the intermolecular interactions in the crystal of the title compound, a Hirshfeld surface (HS) analysis (Spackman & Jayatilaka, 2009) was carried out and the associated two-dimensional fingerprint plots (McKinnon *et al.*, 2007) were generated using *CrystalExplorer17* (Turner *et al.*, 2017). The Hirshfeld surface mapped over d_{norm} using a standard surface resolution with a fixed colour scale of -0.2 (red) to 1.3 (blue) a.u. is shown in Fig. 6a. The shorter and

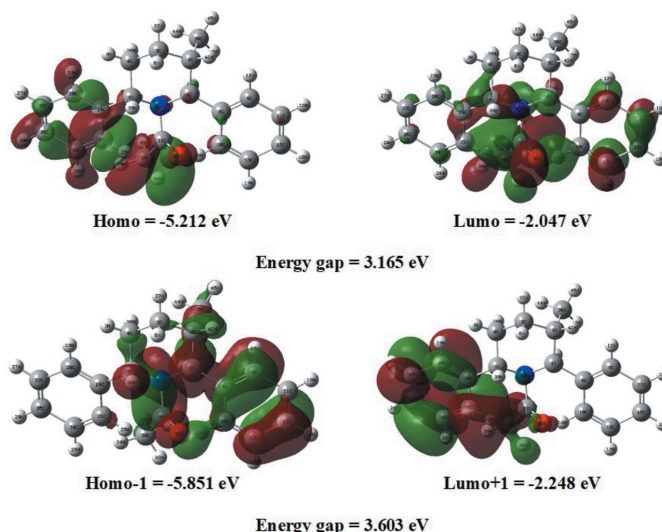


Figure 5
The frontier molecular orbitals (FMOs) of the title compound.

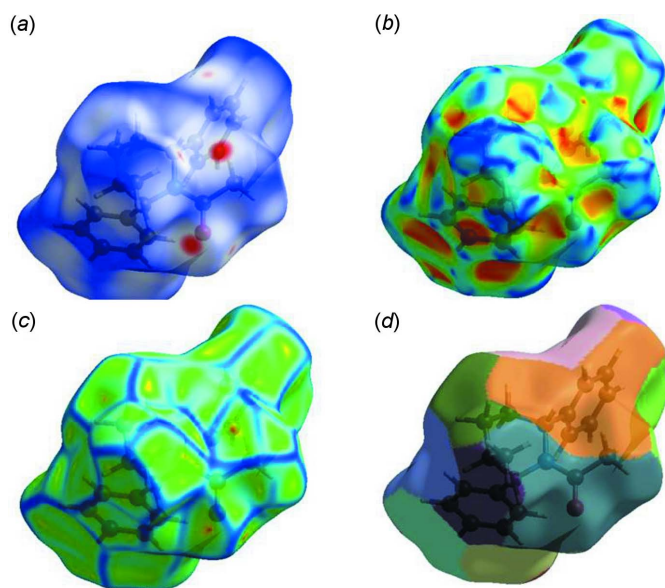


Figure 6
Hirshfeld surfaces mapped over (a) d_{norm} , (b) shape-index, (c) curvedness and (d) fragment patches.

longer contacts are indicated as red and blue spots, respectively, on the Hirshfeld surfaces, and contacts with distances approximately equal to the sum of the van der Waals radii are represented as white spots. The most important red spots on the d_{norm} surface represent C—H...O interactions.

The HS mapped over curvedness and shape-index, introduced by Koendrink (Koenderink, 1990; Koenderink & van

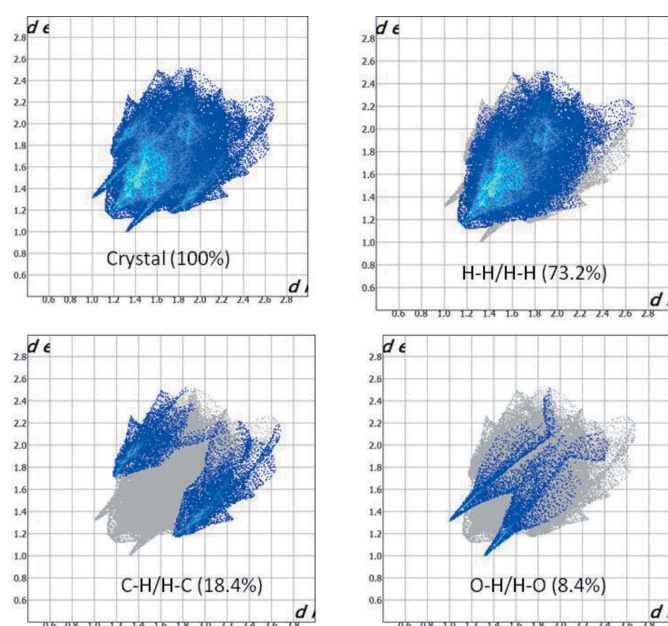


Figure 7
Two-dimensional fingerprint plot for the title compound showing the contributions of individual types of interactions (all intermolecular contacts, H...H contacts, C...H/H...C contacts and O...H/H...O contacts).

Doorn, 1992), give further chemical insight into molecular packing. A surface with low curvedness designates a flat region and may be indicative of π - π stacking in the crystal. A surface with high curvedness is highlighted as dark blue edges, and is indicative of the absence of π - π stacking (Fig. 6). The nearest neighbour coordination environment of a molecule is identified from the colour patches on the Hirshfeld surface, depending on their closeness to adjacent molecules (Mohamooda Sumaya *et al.*, 2017).

The two-dimensional fingerprint plots of (d_i , d_e) points of all the contacts contributing to the Hirshfeld surface analysis in normal mode for all the atoms are shown in Fig. 7. The most important intermolecular interactions are H...H contacts, contributing 73.2% to the overall crystal packing. Other interactions and their respective contributions are C...H/H...C (18.4%) and O...H/H...O (8.4%), respectively.

The Hirshfeld surface analysis confirms the importance of H-atom contacts in establishing the packing. The large number of H...H and C...H/H...C interactions suggest that van der Waals interactions and hydrogen bonding play the major roles in the crystal packing (Hathwar *et al.*, 2015).

6. Database survey

A search of the Cambridge Structural Database (CSD, version 5.39, update August 2018; Groom *et al.*, 2016) for the 3-methyl-2,6-diphenylpiperidine skeleton yielded two hits, methyl 4-oxo-*r*-2,*c*-6-diphenylpiperidine-3-carboxylate (BIHZEY; Sampath *et al.*, 2004) and *r*-2,*c*-6-diphenylpiperidine (NIKYEN; Maheshwaran *et al.*, 2013). The piperidine ring has a boat-shaped conformation in both compounds, as in the title compound. The benzene ring and the mean plane of the piperidine ring are inclined to each other by dihedral angles ranging from 19.95 to 29.16°, compared to 22.05 (6)° in the title compound.

7. Synthesis and crystallization

The compound *t*-3-methyl-*r*-2,*c*-6-diphenylpiperidin-4-one was reduced to the corresponding piperidine using the Wolf-Kishner reduction (Ravindran & Jeyaraman, 1992). The piperidine-4-one (10 mmol) was treated with diethylene glycol (40 ml), hydrazine hydrate (10 mmol) and KOH pellets (10 mmol) to give *t*-3-methyl-*r*-2,*c*-6-diphenylpiperidine. *N*-Acetyl piperidine was synthesized by the acetylation of the above piperidine. To *t*-3-methyl-*r*-2,*c*-6-diphenylpiperidine (5 mmol) dissolved in benzene (50 ml) were added triethylamine (20 mmol) and acetyl chloride (20 mmol) to give *N*-acetyl-*t*-3-methyl-*r*-2,*c*-6-diphenylpiperidine, which was crystallized by slow evaporation from a benzene and petroleum ether solution.

8. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. H atoms were positioned geometrically (N—H = 0.88–0.90 Å and C—H = 0.93–0.98 Å) and

Table 3
Experimental details.

Crystal data	
Chemical formula	C ₂₀ H ₂₃ NO
<i>M</i> _r	293.39
Crystal system, space group	Monoclinic, <i>P</i> 2 ₁
Temperature (K)	296
<i>a</i> , <i>b</i> , <i>c</i> (Å)	8.3063 (4), 7.5842 (4), 13.8410 (7)
β (°)	104.174 (2)
<i>V</i> (Å ³)	845.39 (7)
<i>Z</i>	2
Radiation type	Mo Kα
μ (mm ⁻¹)	0.07
Crystal size (mm)	0.30 × 0.25 × 0.25
Data collection	
Diffractometer	Bruker SMART APEXII CCD
Absorption correction	Multi-scan (<i>SADABS</i> ; Bruker, 2016)
<i>T</i> _{min} , <i>T</i> _{max}	0.697, 0.745
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	15383, 3447, 2821
<i>R</i> _{int}	0.024
(sin θ/λ) _{max} (Å ⁻¹)	0.626
Refinement	
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.037, 0.101, 1.03
No. of reflections	3447
No. of parameters	201
No. of restraints	1
H-atom treatment	H-atom parameters constrained
Δρ _{max} , Δρ _{min} (e Å ⁻³)	0.22, -0.13
Absolute structure	Flack <i>x</i> determined using 1136 quotients [(<i>I</i> ⁺) - (<i>I</i> ⁻)] / [(<i>I</i> ⁺) + (<i>I</i> ⁻)] (Parsons <i>et al.</i> , 2013)
Absolute structure parameter	0.0 (5)

Computer programs: *APEX2* and *SAINT* (Bruker, 2016), *SHELXS97* and *SHELXL97* (Sheldrick, 2008), *SHELXL2018/3* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2020).

allowed to ride on their parent atoms, with *U*_{iso}(H) = 1.5*U*_{eq}(C) for methyl H and 1.2*U*_{eq}(C) for other H atoms.

Acknowledgements

The authors thank the SAIF, IIT Madras, India, for the data collection.

Funding information

KR thanks the UGC, New Delhi, for financial assistance in the form of a Minor Research Project.

References

Aridoss, G., Parthiban, P., Ramachandran, R., Prakash, M., Kabilan, S. & Jeong, Y. T. (2009). *Eur. J. Med. Chem.* **44**, 577–592.
 Beddoes, R. L., Dalton, L., Joule, T. A., Mills, O. S., Street, J. D. & Watt, C. I. F. (1986). *J. Chem. Soc. Perkin Trans. 2*, pp. 787–797.
 Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N. L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Bruker (2016). *APEX3*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
 Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
 Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
 Frisch, M. J., Trucks, G. W., Schlegel, H. B., Scuseria, G. E., Robb, M. A., Cheeseman, J. R., Scalmani, G., Barone, V., Mennucci, B., Petersson, G. A., Nakatsuji, H., Caricato, M., Li, X., Hratchian, H. P., Izmaylov, A. F., Bloino, J., Zheng, G., Sonnenberg, J. L., Hada, M., Ehara, M., Toyota, K., Fukuda, R., Hasegawa, J., Ishida, M., Nakajima, T., Honda, Y., Kitao, O., Nakai, H., Vreven, T., Montgomery, J. A. Jr, Peralta, J. E., Ogliaro, F., Bearpark, M., Heyd, J. J., Brothers, E., Kudin, K. N., Staroverov, V. N., Kobayashi, R., Normand, J., Raghavachari, K., Rendell, A., Burant, J. C., Iyengar, S. S., Tomasi, J., Cossi, M., Rega, N., Millam, J. M., Klene, M., Knox, J. E., Cross, J. B., Bakken, V., Adamo, C., Jaramillo, J., Gomperts, R., Stratmann, R. E., Yazyev, O., Austin, A. J., Cammi, R., Pomelli, C., Ochterski, J. W., Martin, R. L., Morokuma, K., Zakrzewski, V. G., Voth, G. A., Salvador, P., Dannenberg, J. J., Dapprich, S., Daniels, A. D., Farkas, Ö., Foresman, J. B., Ortiz, J. V., Cioslowski, J. & Fox, D. J. (2009). *GAUSSIAN09*. Gaussian Inc., Wallingford, CT, USA.
 Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* **B72**, 171–179.
 Hathwar, V. R., Sist, M., Jørgensen, M. R. V., Mamakhel, A. H., Wang, X., Hoffmann, C. M., Sugimoto, K., Overgaard, J. & Iversen, B. B. (2015). *IUCrJ*, **2**, 563–574.
 Katritzky, A. (2010). *Adv. Heterocycl. Chem.* pp. 42–89.
 Koenderink, J. J. (1990). *Solid Shape*. Cambridge MA: MIT Press.
 Koenderink, J. J. & van Doorn, A. J. (1992). *Image Vis. Comput.* **10**, 557–564.
 Maheshwaran, V., Abdul Basheer, S., Akila, A., Ponnuswamy, S. & Ponnuswamy, M. N. (2013). *Acta Cryst.* **E69**, o1371.
 McKinnon, J. J., Jayatilaka, D. & Spackman, M. A. (2007). *Chem. Commun.* pp. 3814–3816.
 Mobio, I. G., Soldatenkov, A. T., Federov, V. O., Ageev, E. A., Sargeeva, N. D., Lin, S., Stashenko, E. E., Prostakov, N. S. & Andreeva, E. I. (1989). *Khim. Farm. Zh.* **23**, 421–427.
 Mohamooda Sumaya, U., Reuben Jonathan, D., Era, D. T., Gomathi, S. & Usha, G. (2017). *IUCrData*, **2**, x170813.
 Nardelli, M. (1983). *Acta Cryst.* **C39**, 1141–1142.
 Parsons, S., Flack, H. D. & Wagner, T. (2013). *Acta Cryst.* **B69**, 249–259.
 Ravindran, T. & Jeyaraman, R. (1992). *Indian J. Chem.* **B31**, 677–682.
 Roques, R., Declercq, J. P., Germain, G., Graffin, P., Kamenka, J. M. & Geneste, P. (1981). *Acta Cryst.* **B37**, 712–714.
 Sampath, N., Aravindhan, S., Ponnuswamy, M. N. & Nethaji, M. (2004). *Acta Cryst.* **E60**, o2105–o2106.
 Sekar, K., Parthasarathy, S. & Radhakrishnan, T. R. (1990). *Acta Cryst.* **C46**, 1338–1340.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Sheldrick, G. M. (2015). *Acta Cryst.* **C71**, 3–8.
 Spackman, M. A. & Jayatilaka, D. (2009). *CrystEngComm*, **11**, 19–32.
 Spek, A. L. (2020). *Acta Cryst.* **E76**, 1–11.
 Turner, M. J., MacKinnon, J. J., Wolff, S. K., Grimwood, D. J., Spackman, P. R., Jayatilaka, D. & Spackman, M. A. (2017). *Crystal Explorer 17.5*. University of Western Australia.
 Vinaya, K., Kavitha, R., Ananda Kumar, C. S., Benaka Prasad, S. B., Chandrappa, S., Deepak, S. A., NanjundaSwamy, S., Umeha, S. & Rangappa, K. S. (2009). *Arch. Pharm. Res.* **32**, 1, 33–41.

supporting information

Acta Cryst. (2022). E78, 179-183 [https://doi.org/10.1107/S2056989022000275]

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Computing details

Data collection: *APEX2* (Bruker, 2016); cell refinement: *SAINTE* (Bruker, 2016); data reduction: *SAINTE* (Bruker, 2016); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL-2018/3* (Sheldrick, 2015); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2020).

1-(3-Methyl-2,6-diphenylpiperidin-1-yl)ethanone

Crystal data

C₂₀H₂₃NO

M_r = 293.39

Monoclinic, *P*2₁

a = 8.3063 (4) Å

b = 7.5842 (4) Å

c = 13.8410 (7) Å

β = 104.174 (2)°

V = 845.39 (7) Å³

Z = 2

F(000) = 316

D_x = 1.153 Mg m⁻³

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 2821 reflections

θ = 2.6–26.4°

μ = 0.07 mm⁻¹

T = 296 K

Block, white

0.30 × 0.25 × 0.25 mm

Data collection

Bruker SMART APEXII CCD
diffractometer

Radiation source: fine-focus sealed tube

ω and φ scans

Absorption correction: multi-scan
(SADABS; Bruker, 2016)

T_{min} = 0.697, *T_{max}* = 0.745

15383 measured reflections

3447 independent reflections

2821 reflections with *I* > 2σ(*I*)

R_{int} = 0.024

θ_{max} = 26.4°, θ_{min} = 2.6°

h = -10→9

k = -9→9

l = -17→17

Refinement

Refinement on *F*²

Least-squares matrix: full

R[*F*² > 2σ(*F*²)] = 0.037

wR(*F*²) = 0.101

S = 1.03

3447 reflections

201 parameters

1 restraint

Primary atom site location: difference Fourier
map

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

w = 1/[σ²(*F_o*²) + (0.0534*P*)² + 0.0626*P*]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δσ)_{max} < 0.001

Δρ_{max} = 0.22 e Å⁻³

Δρ_{min} = -0.13 e Å⁻³

Absolute structure: Flack x determined using
 1136 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
 Absolute structure parameter: 0.0 (5)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C2	0.3371 (3)	0.2057 (3)	0.31568 (16)	0.0457 (5)
H2	0.377220	0.270737	0.378151	0.055*
C3	0.1580 (3)	0.2626 (4)	0.2699 (2)	0.0565 (6)
H3A	0.155366	0.388579	0.257504	0.068*
H3B	0.117951	0.203547	0.206358	0.068*
C4	0.0435 (3)	0.2199 (4)	0.3367 (2)	0.0679 (7)
H4A	-0.070712	0.231457	0.298638	0.081*
H4B	0.061680	0.304992	0.390657	0.081*
C5	0.0700 (3)	0.0344 (4)	0.38074 (17)	0.0567 (6)
H5	-0.038621	-0.023553	0.368498	0.068*
C6	0.1821 (3)	-0.0774 (3)	0.33117 (17)	0.0481 (5)
H6	0.209751	-0.182736	0.372930	0.058*
C7	0.1060 (3)	-0.1457 (3)	0.22655 (17)	0.0502 (5)
C8	-0.0600 (3)	-0.1247 (4)	0.1781 (2)	0.0659 (7)
H8	-0.129032	-0.058009	0.207568	0.079*
C9	-0.1240 (5)	-0.2032 (5)	0.0855 (2)	0.0812 (10)
H9	-0.235412	-0.188306	0.053477	0.097*
C10	-0.0235 (6)	-0.3020 (5)	0.0415 (2)	0.0890 (11)
H10	-0.066947	-0.354500	-0.020068	0.107*
C11	0.1404 (5)	-0.3236 (4)	0.0881 (2)	0.0824 (10)
H11	0.208456	-0.390605	0.058133	0.099*
C12	0.2053 (4)	-0.2460 (4)	0.1796 (2)	0.0637 (7)
H12	0.317158	-0.260974	0.210498	0.076*
C13	0.4446 (3)	0.2557 (3)	0.24606 (16)	0.0471 (5)
C14	0.4544 (3)	0.1539 (4)	0.16487 (19)	0.0584 (6)
H14	0.395691	0.048462	0.152879	0.070*
C15	0.5502 (4)	0.2063 (5)	0.1012 (2)	0.0715 (8)
H15	0.555322	0.136101	0.047006	0.086*
C16	0.6372 (4)	0.3607 (5)	0.1175 (2)	0.0783 (9)
H16	0.701865	0.395466	0.074701	0.094*
C17	0.6289 (4)	0.4638 (4)	0.1971 (3)	0.0775 (9)
H17	0.688101	0.568970	0.208368	0.093*
C18	0.5326 (3)	0.4126 (4)	0.2611 (2)	0.0632 (7)
H18	0.526974	0.484386	0.314655	0.076*
C19	0.4825 (3)	-0.0674 (3)	0.39016 (16)	0.0476 (5)

C20	0.6396 (3)	0.0380 (4)	0.41982 (19)	0.0617 (7)
H20A	0.724197	-0.031394	0.462980	0.093*
H20B	0.619811	0.142671	0.454141	0.093*
H20C	0.675456	0.070131	0.361322	0.093*
C21	0.1416 (4)	0.0413 (5)	0.4921 (2)	0.0733 (8)
H21A	0.155370	-0.076398	0.518301	0.110*
H21B	0.067488	0.105297	0.522752	0.110*
H21C	0.247396	0.099569	0.506051	0.110*
N1	0.3411 (2)	0.0152 (2)	0.33954 (13)	0.0433 (4)
O1	0.4827 (2)	-0.2245 (3)	0.41220 (16)	0.0707 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C2	0.0422 (11)	0.0485 (13)	0.0457 (11)	0.0023 (10)	0.0097 (9)	-0.0057 (10)
C3	0.0478 (13)	0.0552 (14)	0.0642 (14)	0.0106 (11)	0.0097 (11)	0.0003 (12)
C4	0.0484 (14)	0.0814 (19)	0.0771 (17)	0.0154 (14)	0.0216 (13)	-0.0054 (15)
C5	0.0388 (12)	0.0813 (18)	0.0536 (13)	-0.0051 (12)	0.0180 (10)	-0.0059 (13)
C6	0.0379 (11)	0.0585 (13)	0.0484 (11)	-0.0038 (10)	0.0117 (9)	0.0029 (11)
C7	0.0493 (13)	0.0504 (12)	0.0509 (12)	-0.0083 (11)	0.0123 (10)	0.0014 (11)
C8	0.0556 (15)	0.0738 (19)	0.0623 (15)	-0.0092 (14)	0.0029 (12)	-0.0028 (14)
C9	0.080 (2)	0.085 (2)	0.0654 (17)	-0.0241 (17)	-0.0076 (16)	0.0078 (16)
C10	0.122 (3)	0.086 (2)	0.0541 (16)	-0.035 (2)	0.0132 (19)	-0.0097 (16)
C11	0.109 (3)	0.071 (2)	0.0726 (19)	-0.0196 (18)	0.0327 (19)	-0.0186 (16)
C12	0.0700 (17)	0.0594 (15)	0.0644 (15)	-0.0064 (13)	0.0217 (13)	-0.0078 (13)
C13	0.0420 (12)	0.0468 (12)	0.0502 (12)	0.0015 (10)	0.0068 (9)	0.0034 (10)
C14	0.0599 (14)	0.0624 (15)	0.0547 (14)	-0.0057 (12)	0.0173 (12)	-0.0007 (11)
C15	0.0708 (17)	0.088 (2)	0.0616 (15)	0.0017 (16)	0.0277 (14)	0.0080 (15)
C16	0.0658 (18)	0.092 (2)	0.083 (2)	-0.0003 (17)	0.0287 (15)	0.0310 (19)
C17	0.0677 (17)	0.0639 (18)	0.102 (2)	-0.0122 (14)	0.0221 (17)	0.0219 (17)
C18	0.0586 (15)	0.0526 (14)	0.0755 (16)	-0.0018 (12)	0.0110 (13)	0.0047 (13)
C19	0.0417 (12)	0.0580 (14)	0.0444 (11)	0.0082 (11)	0.0129 (9)	0.0067 (10)
C20	0.0361 (11)	0.0821 (18)	0.0650 (14)	0.0069 (12)	0.0085 (10)	0.0051 (14)
C21	0.0709 (18)	0.091 (2)	0.0638 (15)	-0.0030 (16)	0.0278 (13)	0.0006 (16)
N1	0.0335 (9)	0.0520 (10)	0.0445 (9)	0.0014 (8)	0.0100 (7)	0.0023 (8)
O1	0.0595 (12)	0.0657 (12)	0.0843 (13)	0.0117 (9)	0.0125 (10)	0.0206 (10)

Geometric parameters (Å, °)

C2—N1	1.481 (3)	C11—C12	1.381 (4)
C2—C13	1.513 (3)	C11—H11	0.9300
C2—C3	1.530 (3)	C12—H12	0.9300
C2—H2	0.9800	C13—C14	1.382 (4)
C3—C4	1.515 (4)	C13—C18	1.386 (4)
C3—H3A	0.9700	C14—C15	1.382 (4)
C3—H3B	0.9700	C14—H14	0.9300
C4—C5	1.528 (4)	C15—C16	1.366 (5)
C4—H4A	0.9700	C15—H15	0.9300

C4—H4B	0.9700	C16—C17	1.366 (5)
C5—C21	1.511 (4)	C16—H16	0.9300
C5—C6	1.539 (3)	C17—C18	1.386 (4)
C5—H5	0.9800	C17—H17	0.9300
C6—N1	1.475 (3)	C18—H18	0.9300
C6—C7	1.522 (3)	C19—O1	1.230 (3)
C6—H6	0.9800	C19—N1	1.364 (3)
C7—C8	1.387 (3)	C19—C20	1.499 (4)
C7—C12	1.394 (4)	C20—H20A	0.9600
C8—C9	1.395 (4)	C20—H20B	0.9600
C8—H8	0.9300	C20—H20C	0.9600
C9—C10	1.371 (6)	C21—H21A	0.9600
C9—H9	0.9300	C21—H21B	0.9600
C10—C11	1.366 (5)	C21—H21C	0.9600
C10—H10	0.9300		
N1—C2—C13	113.69 (18)	C10—C11—C12	120.1 (3)
N1—C2—C3	109.49 (19)	C10—C11—H11	119.9
C13—C2—C3	109.34 (19)	C12—C11—H11	119.9
N1—C2—H2	108.1	C11—C12—C7	121.2 (3)
C13—C2—H2	108.1	C11—C12—H12	119.4
C3—C2—H2	108.1	C7—C12—H12	119.4
C4—C3—C2	112.3 (2)	C14—C13—C18	117.9 (2)
C4—C3—H3A	109.2	C14—C13—C2	122.6 (2)
C2—C3—H3A	109.2	C18—C13—C2	119.4 (2)
C4—C3—H3B	109.2	C15—C14—C13	121.0 (3)
C2—C3—H3B	109.2	C15—C14—H14	119.5
H3A—C3—H3B	107.9	C13—C14—H14	119.5
C3—C4—C5	112.9 (2)	C16—C15—C14	120.4 (3)
C3—C4—H4A	109.0	C16—C15—H15	119.8
C5—C4—H4A	109.0	C14—C15—H15	119.8
C3—C4—H4B	109.0	C17—C16—C15	119.6 (3)
C5—C4—H4B	109.0	C17—C16—H16	120.2
H4A—C4—H4B	107.8	C15—C16—H16	120.2
C21—C5—C4	110.9 (2)	C16—C17—C18	120.4 (3)
C21—C5—C6	110.1 (2)	C16—C17—H17	119.8
C4—C5—C6	111.90 (19)	C18—C17—H17	119.8
C21—C5—H5	107.9	C13—C18—C17	120.7 (3)
C4—C5—H5	107.9	C13—C18—H18	119.7
C6—C5—H5	107.9	C17—C18—H18	119.7
N1—C6—C7	113.10 (17)	O1—C19—N1	121.4 (2)
N1—C6—C5	109.31 (19)	O1—C19—C20	120.0 (2)
C7—C6—C5	117.12 (19)	N1—C19—C20	118.5 (2)
N1—C6—H6	105.4	C19—C20—H20A	109.5
C7—C6—H6	105.4	C19—C20—H20B	109.5
C5—C6—H6	105.4	H20A—C20—H20B	109.5
C8—C7—C12	118.0 (2)	C19—C20—H20C	109.5
C8—C7—C6	123.5 (2)	H20A—C20—H20C	109.5

C12—C7—C6	118.3 (2)	H20B—C20—H20C	109.5
C7—C8—C9	120.3 (3)	C5—C21—H21A	109.5
C7—C8—H8	119.8	C5—C21—H21B	109.5
C9—C8—H8	119.8	H21A—C21—H21B	109.5
C10—C9—C8	120.3 (3)	C5—C21—H21C	109.5
C10—C9—H9	119.8	H21A—C21—H21C	109.5
C8—C9—H9	119.8	H21B—C21—H21C	109.5
C11—C10—C9	120.1 (3)	C19—N1—C6	117.61 (18)
C11—C10—H10	120.0	C19—N1—C2	122.16 (19)
C9—C10—H10	120.0	C6—N1—C2	118.46 (17)
N1—C2—C3—C4	56.0 (3)	N1—C2—C13—C18	-141.6 (2)
C13—C2—C3—C4	-178.8 (2)	C3—C2—C13—C18	95.7 (3)
C2—C3—C4—C5	-45.0 (3)	C18—C13—C14—C15	0.6 (4)
C3—C4—C5—C21	112.4 (3)	C2—C13—C14—C15	178.1 (2)
C3—C4—C5—C6	-11.0 (3)	C13—C14—C15—C16	0.0 (5)
C21—C5—C6—N1	-68.0 (3)	C14—C15—C16—C17	-0.2 (5)
C4—C5—C6—N1	55.8 (3)	C15—C16—C17—C18	0.0 (5)
C21—C5—C6—C7	161.7 (2)	C14—C13—C18—C17	-0.8 (4)
C4—C5—C6—C7	-74.5 (3)	C2—C13—C18—C17	-178.4 (2)
N1—C6—C7—C8	-133.9 (2)	C16—C17—C18—C13	0.6 (4)
C5—C6—C7—C8	-5.4 (4)	O1—C19—N1—C6	13.1 (3)
N1—C6—C7—C12	51.0 (3)	C20—C19—N1—C6	-166.3 (2)
C5—C6—C7—C12	179.5 (2)	O1—C19—N1—C2	177.7 (2)
C12—C7—C8—C9	0.3 (4)	C20—C19—N1—C2	-1.7 (3)
C6—C7—C8—C9	-174.8 (3)	C7—C6—N1—C19	-108.2 (2)
C7—C8—C9—C10	0.1 (5)	C5—C6—N1—C19	119.4 (2)
C8—C9—C10—C11	-0.3 (5)	C7—C6—N1—C2	86.6 (2)
C9—C10—C11—C12	0.1 (5)	C5—C6—N1—C2	-45.8 (2)
C10—C11—C12—C7	0.4 (5)	C13—C2—N1—C19	64.0 (3)
C8—C7—C12—C11	-0.6 (4)	C3—C2—N1—C19	-173.40 (18)
C6—C7—C12—C11	174.8 (2)	C13—C2—N1—C6	-131.6 (2)
N1—C2—C13—C14	40.9 (3)	C3—C2—N1—C6	-8.9 (3)
C3—C2—C13—C14	-81.8 (3)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C20—H20B...O1 ⁱ	0.96	2.44	3.292 (3)	148

Symmetry code: (i) $-x+1, y+1/2, -z+1$.