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# Crystal structure and Hirshfeld surface analysis of 3-benzoyl-6-(1,3-dioxo-1-phenylbutan-2-yl)-2-hydroxy-2-methyl-4-phenylcyclohexane-1,1-dicarbonitrile

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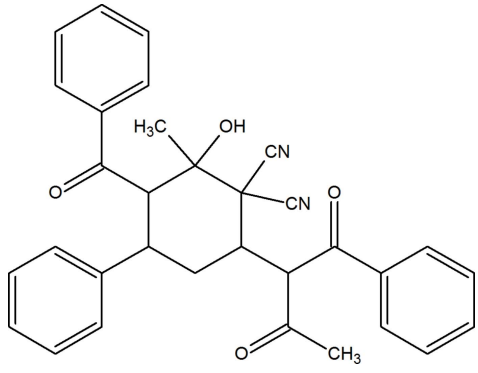
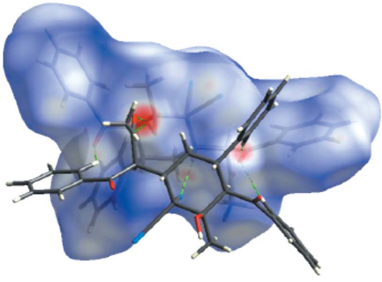
The central cyclohexane ring of the title compound, $C_{32}H_{28}N_2O_4$, adopts a chair conformation, with puckering parameters $Q_T$ = 0.618 (2) Å, $\theta$ = 176.72 (19)° and $\varphi$ = 290 (3)°. In the crystal, molecules are linked by O—H···O, C—H···O and C—H···N hydrogen bonds, forming layers parallel to (100). These layers are linked by weak C—H···$\pi$ interactions and van der Waals forces. A Hirshfeld surface analysis indicates that the contributions from the most prevalent interactions are H···H (41.2% contribution), C···H/H···C (20.3%), O···H/H···O (17.8%) and N···H/H···N (10.6%).

## 1. Chemical context

Functionalized derivatives of carbo- and heterocyclic compounds are of great interest in the fields of organic synthesis, catalysis, materials science and medicinal chemistry (Zubkov *et al.*, 2018; Shikhaliyev *et al.*, 2019; Viswanathan *et al.*, 2019; Gurbanov *et al.*, 2020; Khalilov *et al.*, 2021). In particular, $\beta$-dicarbonyl compounds are important chemical substrates for the construction of various classes of organic compounds (Kaur *et al.*, 2021).




To the best of our knowledge, the interaction of $\beta$-dicarbonyl compounds with phenyl–allylidene–malononitriles leads to the formation of xanthene, benzo[*b*]pyran and pyridine derivatives (Bardasov *et al.*, 2014; Amoozadeh *et al.*, 2018). Interestingly, we discovered that in case of the reaction of one equivalent of phenyl–allylidene–malononitrile with two



equivalents of benzoylacetone at room temperature, a substituted cyclohexane derivative was the product. In the context of ongoing structural studies (Safavora *et al.*, 2019; Aliyeva *et al.*, 2011; Mamedov *et al.*, 2022), we report here the crystal structure and Hirshfeld surface analysis of the title compound, 3-benzoyl-6-(1,3-dioxo-1-phenylbutan-2-yl)-2-hydroxy-2-methyl-4-phenylcyclohexane-1,1-dicarbonitrile.

## 2. Structural commentary

In the title compound (Fig. 1), the central cyclohexane ring (*A*: atoms C1–C6) adopts a chair conformation, with puckering parameters (Cremer & Pople, 1975) $Q_T$ = 0.618 (2) Å, $\theta$ = 176.72 (19)° and $\varphi$ = 290 (3)°. The phenyl (*B*: C11–C16; *C*: C21–C26; *D*: C27–C32) rings make dihedral angles of 78.23 (10), 83.20 (11) and 82.09 (10)°, respectively, with the mean plane of the central cyclohexane ring. The dihedral angles between the phenyl rings are *B*/*C* = 21.88 (10)°, *B*/*D* = 21.88 (19)° and *C*/*D* = 73.64 (10)°. The C1—C7—C10—C11, C1—C7—C10—O2, C1—C7—C8—C9 and C1—C7—C8—O1 torsion angles are −157.13 (16), 27.9 (2), −73.6 (2) and 106.7 (2)°. The phenyl, benzoyl, hydroxy, cyano C2—C17≡N1 and 1,3-dioxo-1-phenylbutan-2-yl substituents all occupy equatorial sites, so that the cyano C2—C18≡N2 substituent necessarily occupies an axial site. There are five stereogenic centres and the chirality about the C1, C3, C4, C5 and C7 atoms are *S*, *R*, *R*, *S* and *R*, respectively. The values of the geometric parameters of the title compound are normal and compatible with those of related compounds compiled in the *Database survey* section (§5).

## 3. Supramolecular features

In the crystal, O—H···O hydrogen bonds of medium strength, and weaker C—H···O and C—H···N interactions link adjacent molecules, forming layers extending parallel to (100) (Table 1 and Figs. 2–4). These layers are connected by weak C—H···$\pi$ interactions and van der Waals interactions (Table 1 and Fig. 5).

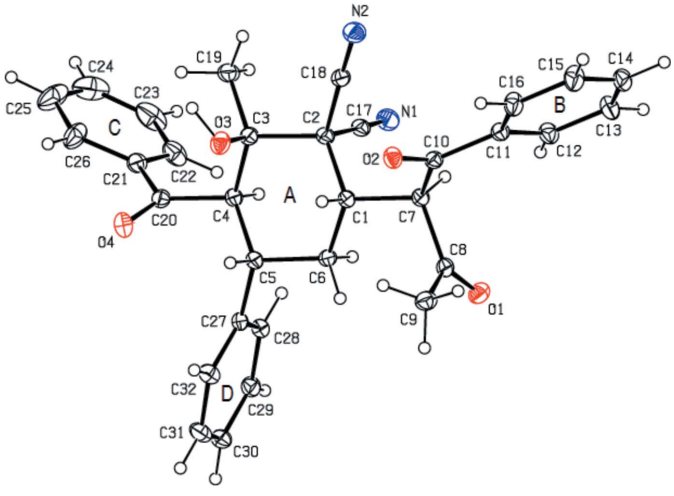



**Figure 1**
The molecular structure of the title compound, showing the labelling scheme and displacement ellipsoids drawn at the 30% probability level.

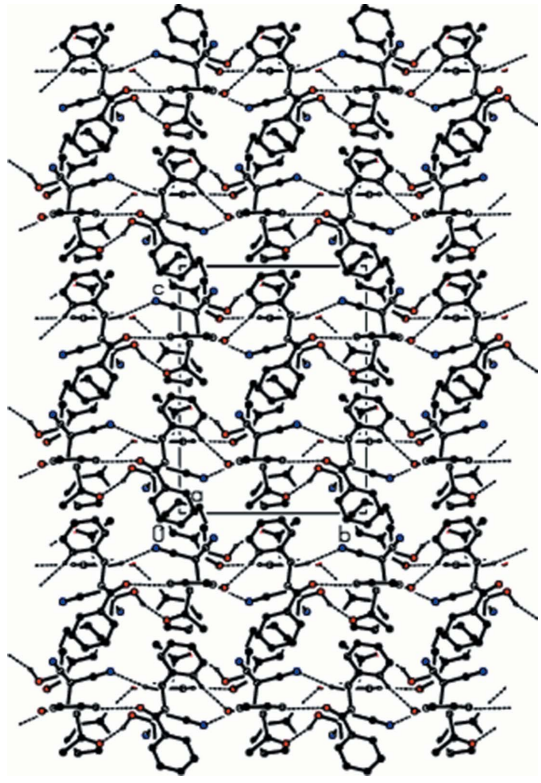



**Figure 2**
A view of the molecular packing down [100], showing O—H···O, C—H···O and C—H···N hydrogen bonds as dashed lines.

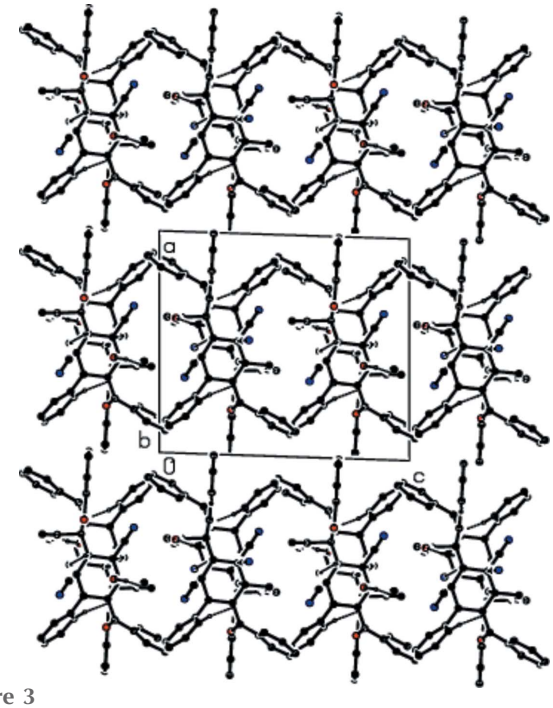



**Figure 3**
A view of the molecular packing down [010]. Intermolecular interactions are depicted as in Fig. 2.

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

*Cg*4 is the centroid of the C27–C32 phenyl ring.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O3—H3···O1$^{i}$ | 0.94 (3) | 1.89 (3) | 2.787 (2) | 159 (3) |
| C1—H1···N1$^{i}$ | 1.00 | 2.50 | 3.466 (3) | 163 |
| C12—H12···O4$^{ii}$ | 0.95 | 2.58 | 3.242 (3) | 127 |
| C28—H28···O2$^{ii}$ | 0.95 | 2.40 | 3.319 (2) | 164 |
| C14—H14···*Cg*4$^{iii}$ | 0.95 | 2.80 | 3.475 (2) | 129 |

Symmetry codes: (i) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$; (ii) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$; (iii) $x+1, y, z$.

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

| Contact | Distance | Symmetry operation |
|---|---|---|
| O1···H3 | 1.89 | $1-x, \frac{1}{2}+y, \frac{1}{2}-z$ |
| H9*A*···H6*A* | 2.39 | $1-x, 1-y, -z$ |
| O4···H15 | 2.73 | $-1+x, y, z$ |
| H19*B*···N1 | 2.77 | $1-x, 1-y, 1-z$ |
| H26···H31 | 2.37 | $x, \frac{1}{2}-y, \frac{1}{2}+z$ |
| C25···C24 | 3.367 | $-x, 1-y, 1-z$ |
| H29···H23 | 2.41 | $x, \frac{3}{2}-y, -\frac{1}{2}+z$ |
| H13···H15 | 2.36 | $2-x, \frac{1}{2}+y, \frac{1}{2}-z$ |

## 4. Hirshfeld surface analysis

A Hirshfeld surface for the title compound and its associated two-dimensional fingerprint plots were analyzed and calculated using *CrystalExplorer* (Version 17.5; Turner *et al.*, 2017). Hirshfeld surfaces allow for the display of intermolecular interactions by using distinct colours and intensities to indicate short and long contacts, as well as the relative strengths of the interactions. The three-dimensional (3D) Hirshfeld surface of the title compound plotted over $d_{\rm norm}$ in the range from −0.5877 to +1.7202 a.u. is shown in Fig. 6. As discussed above, the O3—H3···O1 interactions play a key role in the molecular packing of the title compound.

The overall two-dimensional (2D) fingerprint plot [Fig. 7(*a*)] and those delineated into H···H (41.2% contribution), C···H/H···C (20.3%), O···H/H···O (17.8%) and N···H/H···N (10.6%) contacts are illustrated in Figs. 7(*b*)–(*e*), respectively. The other minor contributions to the Hirshfeld surface are from N···C/C···N (1.0%), C···C (0.9%), O···N/N···O (0.8%) and O···C/C···O (0.8%) contacts. The large number of H···H, C···H/H···C, O···H/H···O and N···H/H···N interactions suggest that van der Waals interactions and hydrogen bonding play major roles in the crystal packing. Various interatomic contacts are compiled in Table 2.

## 5. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.42, update of September 2021; Groom *et al.*, 2016) for the 2-hydroxy-2-methylcyclohexane-1,1-dicarbonitrile moiety revealed five structures closely related to the title compound: 3-cyano-4-hydroxy-2-(4-methylphenyl)-6-oxo-*N*-phenyl-4-(thiophen-2-yl)cyclohexane-1-carboxamide hydrate (CSD refcode UPOMOE; Naghiyev *et al.*, 2021), (2*RS*,3*SR*,4*RS*,6*SR*)-3-benzoyl-4-hydroxy-2,4,6-triphenylcyclohexane-1,1-dicarbonitrile (MEHMOC01; Rodríguez *et al.*, 2008), 3-(4-fluorobenzoyl)-4-(4-fluorophenyl)-4-hydroxy-2,6-diphenylcyclohexane-1,1-dicarbonitrile (SODHAW; Narayana *et al.*, 2014), 5-cyano-2-hydroxy-2-methyl-*N*-phenyl-4-(pyridin-4-yl)-6-(thiophen-2-yl)-3,4-dihydro-2*H*-pyran-3-carboxamide (JUPHUA; Naghiyev *et al.*, 2020) and 5-cyano-2-hydroxy-2-methyl-6-oxo-*N*-phenyl-4-(thiophen-2-yl)piperi-

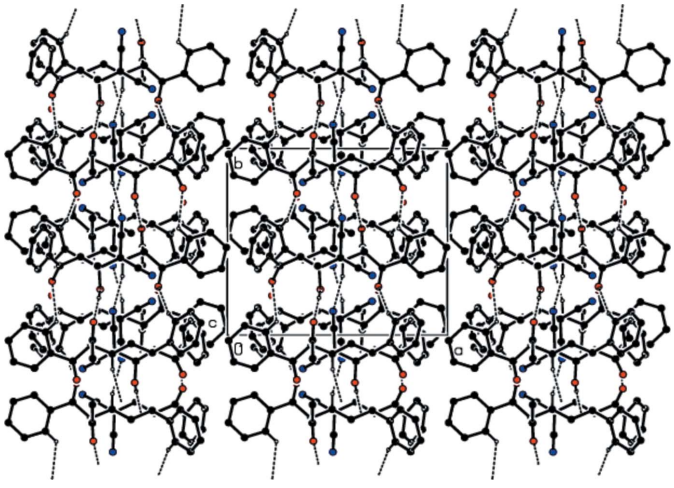


**Figure 4**
A view of the molecular packing down [001]. Intermolecular interactions are depicted as in Fig. 2.

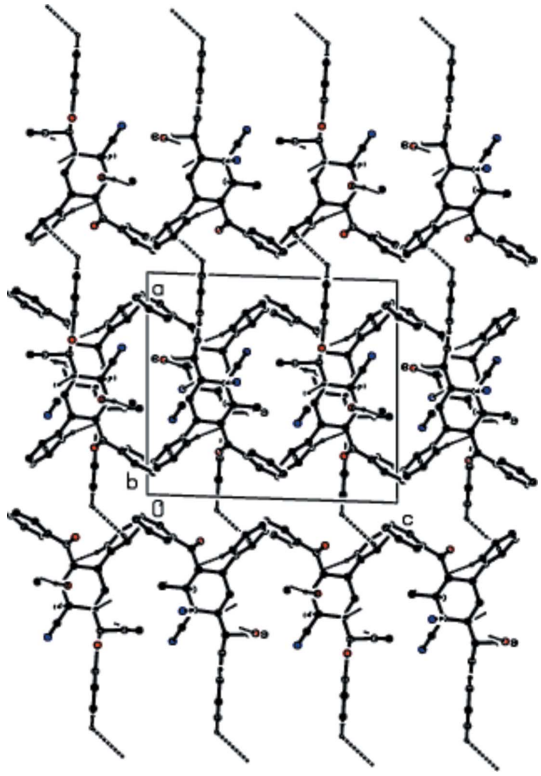


**Figure 5**
The crystal packing viewed down [010], showing O—H···O, C—H···O, C—H···N hydrogen bonds and C—H···$\pi$ interactions.

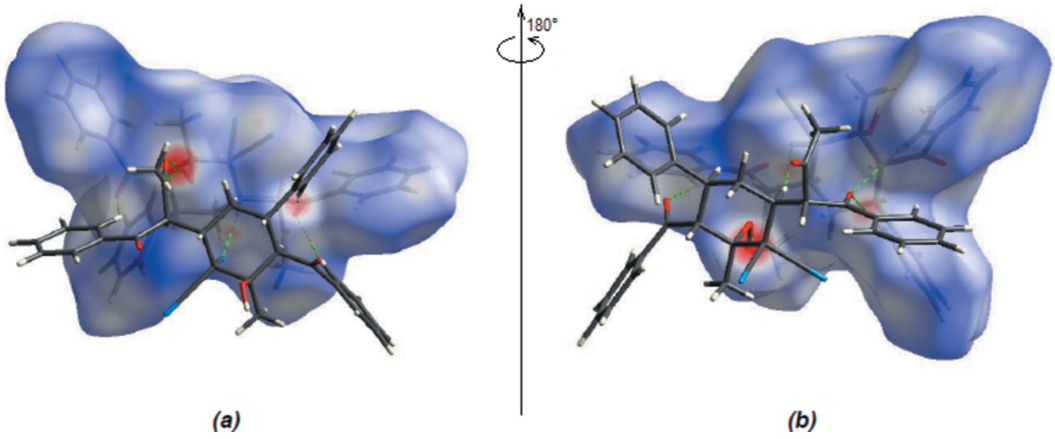



**Figure 6**
(*a*) Front and (*b*) back sides of the 3D Hirshfeld surface of the title compound plotted over $d_{norm}$ in the range from −0.5877 to +1.7202 a.u.

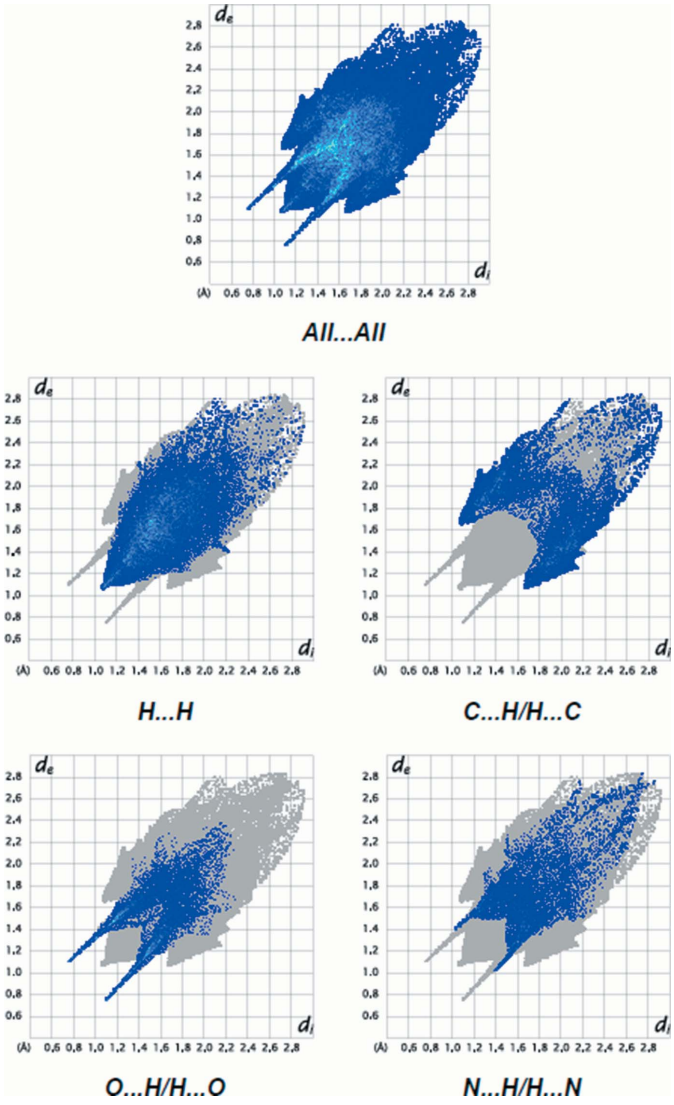



**Figure 7**
A view of the 2D fingerprint plots for the title compound, showing (*a*) all interactions, and delineated into (*b*) H···H, (*c*) C···H/H···C, (*d*) O···H/H···O and (*e*) N···H/H···N interactions. The $d_i$ and $d_e$ values are the closest internal and external distances (in Å) from given points on the Hirshfeld surface.

dine-3-carboxamide methanol solvate (JUPJOW; Naghiyev *et al.*, 2020).

In the crystal of UPOMOE, the central cyclohexane ring adopts a chair conformation. Molecules are linked by N—H···O, C—H···O and C—H···N hydrogen bonds, forming layers parallel to (100), which interact *via* the van der Waals forces between them.

In the crystal of MEHMOC01, the molecules are linked into complex sheets by two C—H···O hydrogen bonds and three C—H···N hydrogen bonds.

In the crystal of SODHAW, molecules are linked *via* pairs of O—H···N hydrogen bonds, forming inversion dimers. The dimers are linked *via* C—H···N and C—H···O hydrogen bonds, forming chains parallel to [001]. C—H···F hydrogen bonds link the chains into sheets lying parallel to (100).

In JUPHUA, the crystal structure is stabilized by an extensive hydrogen-bonding network defined by N—H···N, O—H···N and C—H···O interactions with graph-set motifs $C(9)$, $C(8)$, $C_4^4(32)$ and $R_6^6(48)$, with base vectors [100], [011] and [110] for the 3D network.

In JUPJOW, the crystal structure is also stabilized by an extensive hydrogen-bonding network of N—H···O, O—H···O and O—H···N interactions, where the methanol molecule participate with neighbouring molecules with graph-set motifs $C(4)$, $C_2^2(10)$, $C_4^4(28)$, $R_2^2(8)$ and $R_6^6(36)$, with base vectors [010], [100] and [001] for the 3D network. For JUPHUA and JUPJOW, another non-covalent weak interaction is also observed, specifically a chalcogen···$\pi$ interaction (*ca* 3.6 Å) in JUPHUA between the thiophenyl sulfur fragment and the phenyl ring and a hydrogen···$\pi$ interaction (*ca* 3.2 Å) in JUPJOW between the methyl group on the piperidone ring and the phenyl ring.

## 6. Synthesis and crystallization

To a solution of 2-(3-phenylallylidene)malononitrile (0.92 g, 5 mmol) and benzoylacetone (1.68 g, 10 mmol) in benzene (25 ml), 3–4 drops of 1-methylpiperazine were added and the mixture was stirred for 10 min and kept at room temperature

Table 3
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $C_{32}H_{28}N_2O_4$ |
| $M_r$ | 504.56 |
| Crystal system, space group | Monoclinic, $P2_1/c$ |
| Temperature (K) | 100 |
| $a$, $b$, $c$ (Å) | 13.9798 (3), 11.8411 (2), 15.7406 (3) |
| $\beta$ (°) | 91.901 (2) |
| $V$ (Å$^3$) | 2604.21 (9) |
| $Z$ | 4 |
| Radiation type | Cu $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.69 |
| Crystal size (mm) | 0.09 × 0.06 × 0.06 |
| Data collection | |
| Diffractometer | Rigaku XtaLAB Synergy Dualflex HyPix |
| Absorption correction | Multi-scan (*CrysAlis PRO*; Rigaku OD, 2021) |
| $T_{min}$, $T_{max}$ | 0.933, 0.949 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 77914, 5618, 5497 |
| $R_{int}$ | 0.110 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.639 |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.074, 0.187, 1.11 |
| No. of reflections | 5618 |
| No. of parameters | 348 |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.54, −0.33 |

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

for 72 h. Benzene (15 ml) was then removed from the reaction mixture by distillation, which was left overnight. The crystals which formed were separated by filtration and recrystallized from an ethanol–water (1:1 *v*/*v*) solution (yield 41%; m.p. 514–515 K).

$^1$H NMR (300 MHz, DMSO-$d_6$, ppm): $\delta$ 1.74 (*s*, 3H, $CH_3$), 2.01 (*t*, 2H, $CH_2$), 2.12 (*s*, 3H, $COCH_3$), 3.47 (*d*–*d*, 1H, CH), 3.52 (*s*, 1H, OH), 4.08 (*m*, 1H, CH), 4.62 (*d*, 1H, CH), 4.86 (*d*, 1H, CH), 7.12–7.78 (*m*, 15H, 15Ar-H). $^{13}$C NMR (75 MHz, DMSO-$d_6$, ppm): $\delta$ 24.28 ($CH_3$), 30.36 ($COCH_3$), 34.42 ($CH_2$), 39.41 (CH), 45.49 (CH), 56.46 ($C_{tert}$), 57.01 (CH), 60.85 (CH), 81.92 (O—$C_{tert}$), 111.37 (CN), 111.81 (CN), 125.94 ($CH_{arom}$), 127.22 ($2CH_{arom}$), 127.86 ($2CH_{arom}$), 128.90 ($2CH_{arom}$), 128.98 ($2CH_{arom}$), 129.31 ($2CH_{arom}$), 130.35 ($2CH_{arom}$), 132.52 ($CH_{arom}$), 133.85 ($CH_{arom}$), 135.44 ($C_{arom}$), 138.49 ($C_{arom}$), 141.22 ($C_{arom}$), 194.97 (C═O), 195.93 (C═O), 200.21 (C═O).

## 7. Refinement details

Crystal data, data collection and structure refinement details are summarized in Table 3. Due to large differences between calculated and observed intensities, about 40 reflections were omitted from the refinement. The H atom of the OH group was located in a difference map and its positional parameters were allowed to refine freely [O3—H3 = 0.93 (3) Å], with $U_{iso}$(H) = 1.5$U_{eq}$(O). All H atoms bound to C atoms were positioned geometrically and refined as riding, with C—H = 0.95 (aromatic), 0.99 (methylene), 1.00 (methine) and 0.98 Å (methyl), with $U_{iso}$(H) = 1.5$U_{eq}$(C) for methyl H atoms and 1.2$U_{eq}$(C) for the others.

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# supporting information

*Acta Cryst.* (2022). E**78**, 568-573 [https://doi.org/10.1107/S2056989022004777]

## Crystal structure and Hirshfeld surface analysis of 3-benzoyl-6-(1,3-dioxo-1-phenylbutan-2-yl)-2-hydroxy-2-methyl-4-phenylcyclohexane-1,1-dicarbonitrile

**Farid N. Naghiyev, Victor N. Khrustalev, Ekaterina V. Dobrokhotova, Mehmet Akkurt, Ali N. Khalilov, Ajaya Bhattarai and İbrahim G. Mamedov**

### 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).

### 3-Benzoyl-6-(1,3-dioxo-1-phenylbutan-2-yl)-2-hydroxy-2-methyl-4-phenylcyclohexane-1,1-dicarbonitrile

*Crystal data*

$C_{32}H_{28}N_2O_4$
$M_r$ = 504.56
Monoclinic, $P2_1/c$
$a$ = 13.9798 (3) Å
$b$ = 11.8411 (2) Å
$c$ = 15.7406 (3) Å
$\beta$ = 91.901 (2)°
$V$ = 2604.21 (9) Å$^3$
$Z$ = 4
$F(000)$ = 1064
$D_x$ = 1.287 Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda$ = 1.54184 Å
Cell parameters from 52541 reflections
$\theta$ = 3.1–79.4°
$\mu$ = 0.69 mm$^{-1}$
$T$ = 100 K
Prism, colourless
0.09 × 0.06 × 0.06 mm

*Data collection*

Rigaku XtaLAB Synergy Dualflex HyPix diffractometer
Radiation source: micro-focus sealed X-ray tube
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2021)
$T_{min}$ = 0.933, $T_{max}$ = 0.949
77914 measured reflections
5618 independent reflections
5497 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.110
$\theta_{max}$ = 80.3°, $\theta_{min}$ = 4.7°
$h$ = −17→17
$k$ = −14→12
$l$ = −20→20

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.074
$wR(F^2)$ = 0.187
$S$ = 1.11
5618 reflections
348 parameters
0 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

$w = 1/[\sigma^2(F_o^2) + (0.0869P)^2 + 2.2708P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.54$ e Å$^{-3}$
$\Delta\rho_{min} = -0.33$ e Å$^{-3}$

*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 (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.60930 (11) | 0.56945 (13) | 0.06669 (10) | 0.0327 (3) |
| O2 | 0.68557 (10) | 0.25976 (12) | 0.19898 (9) | 0.0290 (3) |
| O3 | 0.42148 (11) | 0.24428 (13) | 0.31883 (10) | 0.0324 (3) |
| H3 | 0.415 (2) | 0.199 (3) | 0.367 (2) | 0.049* |
| O4 | 0.20303 (11) | 0.28810 (13) | 0.28805 (10) | 0.0351 (4) |
| N1 | 0.52004 (13) | 0.62769 (17) | 0.35886 (12) | 0.0344 (4) |
| N2 | 0.65726 (13) | 0.31942 (18) | 0.39833 (12) | 0.0368 (4) |
| C1 | 0.52388 (13) | 0.39859 (16) | 0.21623 (12) | 0.0231 (4) |
| H1 | 0.5225 | 0.3158 | 0.2038 | 0.028* |
| C2 | 0.51310 (13) | 0.41460 (17) | 0.31391 (12) | 0.0240 (4) |
| C3 | 0.41622 (13) | 0.35964 (18) | 0.34400 (12) | 0.0266 (4) |
| C4 | 0.33213 (13) | 0.42000 (17) | 0.29384 (12) | 0.0237 (4) |
| H4 | 0.3342 | 0.5025 | 0.3074 | 0.028* |
| C5 | 0.34164 (13) | 0.40464 (16) | 0.19736 (12) | 0.0236 (4) |
| H5 | 0.3408 | 0.3219 | 0.1846 | 0.028* |
| C6 | 0.43762 (13) | 0.45324 (17) | 0.16959 (12) | 0.0244 (4) |
| H6A | 0.4432 | 0.4417 | 0.1077 | 0.029* |
| H6B | 0.4386 | 0.5356 | 0.1805 | 0.029* |
| C7 | 0.62107 (13) | 0.44637 (16) | 0.18714 (12) | 0.0237 (4) |
| H7 | 0.6368 | 0.5172 | 0.2193 | 0.028* |
| C8 | 0.61386 (13) | 0.47219 (17) | 0.09111 (12) | 0.0261 (4) |
| C9 | 0.61333 (15) | 0.3741 (2) | 0.03178 (13) | 0.0320 (4) |
| H9A | 0.5778 | 0.3941 | −0.0208 | 0.048* |
| H9B | 0.5825 | 0.3095 | 0.0585 | 0.048* |
| H9C | 0.6793 | 0.3543 | 0.0187 | 0.048* |
| C10 | 0.70293 (13) | 0.36013 (16) | 0.20056 (12) | 0.0235 (4) |
| C11 | 0.80373 (13) | 0.40261 (17) | 0.20804 (12) | 0.0255 (4) |
| C12 | 0.82669 (14) | 0.51657 (19) | 0.20982 (14) | 0.0308 (4) |
| H12 | 0.7773 | 0.5717 | 0.2078 | 0.037* |
| C13 | 0.92204 (15) | 0.5500 (2) | 0.21452 (15) | 0.0352 (5) |
| H13 | 0.9376 | 0.6281 | 0.2152 | 0.042* |
| C14 | 0.99465 (15) | 0.4702 (2) | 0.21823 (15) | 0.0364 (5) |
| H14 | 1.0597 | 0.4935 | 0.2220 | 0.044* |
| C15 | 0.97192 (15) | 0.3563 (2) | 0.21636 (17) | 0.0403 (6) |
| H15 | 1.0215 | 0.3015 | 0.2188 | 0.048* |
| C16 | 0.87744 (15) | 0.32241 (19) | 0.21094 (15) | 0.0346 (5) |

| | | | | |
|---|---|---|---|---|
| H16 | 0.8623 | 0.2442 | 0.2092 | 0.041* |
| C17 | 0.51666 (13) | 0.53547 (18) | 0.33812 (12) | 0.0257 (4) |
| C18 | 0.59353 (14) | 0.35950 (18) | 0.36132 (12) | 0.0269 (4) |
| C19 | 0.40934 (15) | 0.3705 (2) | 0.43962 (13) | 0.0343 (5) |
| H19A | 0.4054 | 0.4505 | 0.4550 | 0.051* |
| H19B | 0.4662 | 0.3368 | 0.4675 | 0.051* |
| H19C | 0.3520 | 0.3311 | 0.4581 | 0.051* |
| C20 | 0.23709 (13) | 0.37142 (17) | 0.32275 (12) | 0.0254 (4) |
| C21 | 0.19117 (14) | 0.42334 (19) | 0.39784 (13) | 0.0301 (4) |
| C22 | 0.20292 (16) | 0.5357 (2) | 0.41997 (16) | 0.0403 (5) |
| H22 | 0.2385 | 0.5844 | 0.3849 | 0.048* |
| C23 | 0.16292 (19) | 0.5777 (3) | 0.4932 (2) | 0.0582 (8) |
| H23 | 0.1697 | 0.6553 | 0.5074 | 0.070* |
| C24 | 0.1136 (2) | 0.5067 (4) | 0.54509 (18) | 0.0669 (10) |
| H24 | 0.0878 | 0.5351 | 0.5959 | 0.080* |
| C25 | 0.1012 (2) | 0.3952 (4) | 0.5241 (2) | 0.0663 (10) |
| H25 | 0.0675 | 0.3465 | 0.5606 | 0.080* |
| C26 | 0.13807 (17) | 0.3531 (3) | 0.44923 (17) | 0.0457 (6) |
| H26 | 0.1270 | 0.2767 | 0.4333 | 0.055* |
| C27 | 0.26091 (13) | 0.46041 (17) | 0.14557 (12) | 0.0243 (4) |
| C28 | 0.23681 (13) | 0.57302 (17) | 0.15741 (13) | 0.0261 (4) |
| H28 | 0.2681 | 0.6150 | 0.2016 | 0.031* |
| C29 | 0.16717 (14) | 0.62547 (19) | 0.10523 (13) | 0.0297 (4) |
| H29 | 0.1507 | 0.7022 | 0.1147 | 0.036* |
| C30 | 0.12222 (14) | 0.5654 (2) | 0.03973 (14) | 0.0330 (5) |
| H30 | 0.0756 | 0.6011 | 0.0035 | 0.040* |
| C31 | 0.14567 (15) | 0.4533 (2) | 0.02747 (14) | 0.0346 (5) |
| H31 | 0.1149 | 0.4119 | −0.0173 | 0.042* |
| C32 | 0.21395 (15) | 0.40056 (19) | 0.08018 (13) | 0.0301 (4) |
| H32 | 0.2287 | 0.3232 | 0.0716 | 0.036* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0331 (8) | 0.0336 (8) | 0.0315 (8) | 0.0030 (6) | 0.0039 (6) | 0.0075 (6) |
| O2 | 0.0234 (7) | 0.0273 (7) | 0.0364 (8) | 0.0000 (5) | 0.0006 (5) | 0.0033 (6) |
| O3 | 0.0323 (8) | 0.0267 (7) | 0.0385 (8) | −0.0003 (6) | 0.0031 (6) | 0.0060 (6) |
| O4 | 0.0280 (7) | 0.0334 (8) | 0.0444 (9) | −0.0061 (6) | 0.0048 (6) | −0.0021 (7) |
| N1 | 0.0320 (9) | 0.0354 (10) | 0.0355 (9) | 0.0023 (7) | −0.0010 (7) | −0.0007 (8) |
| N2 | 0.0290 (9) | 0.0458 (11) | 0.0353 (10) | 0.0044 (8) | −0.0025 (7) | 0.0073 (8) |
| C1 | 0.0188 (8) | 0.0272 (9) | 0.0234 (9) | −0.0002 (7) | 0.0000 (6) | 0.0028 (7) |
| C2 | 0.0198 (8) | 0.0288 (10) | 0.0233 (9) | 0.0014 (7) | −0.0017 (7) | 0.0021 (7) |
| C3 | 0.0215 (9) | 0.0329 (10) | 0.0255 (9) | 0.0000 (7) | 0.0005 (7) | 0.0063 (8) |
| C4 | 0.0197 (8) | 0.0279 (9) | 0.0236 (9) | 0.0002 (7) | 0.0009 (7) | 0.0011 (7) |
| C5 | 0.0195 (8) | 0.0276 (9) | 0.0236 (9) | −0.0009 (7) | −0.0003 (6) | 0.0014 (7) |
| C6 | 0.0207 (8) | 0.0319 (10) | 0.0204 (8) | 0.0006 (7) | 0.0006 (6) | 0.0034 (7) |
| C7 | 0.0193 (8) | 0.0270 (9) | 0.0248 (9) | 0.0007 (7) | 0.0002 (6) | 0.0028 (7) |
| C8 | 0.0166 (8) | 0.0338 (10) | 0.0280 (9) | 0.0010 (7) | 0.0026 (6) | 0.0046 (8) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C9 | 0.0304 (10) | 0.0407 (12) | 0.0250 (9) | 0.0034 (8) | 0.0017 (8) | 0.0022 (8) |
| C10 | 0.0209 (8) | 0.0272 (9) | 0.0225 (8) | 0.0020 (7) | 0.0011 (6) | 0.0030 (7) |
| C11 | 0.0199 (8) | 0.0318 (10) | 0.0248 (9) | 0.0022 (7) | 0.0009 (7) | 0.0029 (7) |
| C12 | 0.0230 (9) | 0.0331 (11) | 0.0361 (11) | 0.0012 (8) | −0.0006 (7) | 0.0009 (8) |
| C13 | 0.0245 (10) | 0.0347 (11) | 0.0462 (12) | −0.0045 (8) | −0.0024 (8) | −0.0007 (9) |
| C14 | 0.0193 (9) | 0.0457 (13) | 0.0441 (12) | −0.0019 (8) | −0.0011 (8) | 0.0112 (10) |
| C15 | 0.0216 (10) | 0.0410 (13) | 0.0585 (15) | 0.0064 (8) | 0.0042 (9) | 0.0166 (11) |
| C16 | 0.0228 (9) | 0.0328 (11) | 0.0482 (13) | 0.0028 (8) | 0.0027 (8) | 0.0112 (9) |
| C17 | 0.0196 (8) | 0.0335 (11) | 0.0238 (9) | 0.0016 (7) | −0.0016 (6) | 0.0025 (7) |
| C18 | 0.0235 (9) | 0.0335 (10) | 0.0235 (9) | 0.0007 (7) | 0.0006 (7) | 0.0045 (8) |
| C19 | 0.0251 (10) | 0.0536 (13) | 0.0242 (10) | 0.0017 (9) | 0.0007 (7) | 0.0010 (9) |
| C20 | 0.0226 (9) | 0.0269 (9) | 0.0266 (9) | 0.0015 (7) | 0.0002 (7) | 0.0047 (7) |
| C21 | 0.0187 (8) | 0.0427 (12) | 0.0289 (10) | 0.0045 (8) | 0.0020 (7) | 0.0038 (8) |
| C22 | 0.0237 (10) | 0.0520 (14) | 0.0453 (13) | 0.0038 (9) | 0.0017 (9) | −0.0153 (11) |
| C23 | 0.0311 (12) | 0.088 (2) | 0.0552 (17) | 0.0090 (13) | 0.0003 (11) | −0.0338 (16) |
| C24 | 0.0444 (15) | 0.121 (3) | 0.0360 (13) | 0.0343 (18) | 0.0033 (11) | −0.0123 (16) |
| C25 | 0.0412 (15) | 0.112 (3) | 0.0469 (16) | 0.0273 (16) | 0.0208 (12) | 0.0340 (18) |
| C26 | 0.0336 (12) | 0.0575 (16) | 0.0470 (14) | 0.0113 (10) | 0.0145 (10) | 0.0213 (12) |
| C27 | 0.0181 (8) | 0.0312 (10) | 0.0236 (9) | −0.0027 (7) | 0.0015 (6) | 0.0025 (7) |
| C28 | 0.0222 (9) | 0.0288 (10) | 0.0271 (9) | −0.0025 (7) | −0.0007 (7) | 0.0017 (7) |
| C29 | 0.0236 (9) | 0.0322 (10) | 0.0333 (10) | 0.0007 (8) | 0.0002 (7) | 0.0045 (8) |
| C30 | 0.0225 (9) | 0.0446 (12) | 0.0315 (10) | 0.0006 (8) | −0.0037 (7) | 0.0076 (9) |
| C31 | 0.0267 (10) | 0.0446 (12) | 0.0321 (10) | −0.0014 (9) | −0.0076 (8) | −0.0044 (9) |
| C32 | 0.0261 (9) | 0.0340 (11) | 0.0300 (10) | 0.0012 (8) | −0.0023 (8) | −0.0039 (8) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C8 | 1.215 (3) | C12—H12 | 0.9500 |
| O2—C10 | 1.213 (2) | C13—C14 | 1.387 (3) |
| O3—C3 | 1.425 (3) | C13—H13 | 0.9500 |
| O3—H3 | 0.93 (3) | C14—C15 | 1.386 (4) |
| O4—C20 | 1.217 (3) | C14—H14 | 0.9500 |
| N1—C17 | 1.140 (3) | C15—C16 | 1.380 (3) |
| N2—C18 | 1.151 (3) | C15—H15 | 0.9500 |
| C1—C6 | 1.534 (2) | C16—H16 | 0.9500 |
| C1—C7 | 1.555 (2) | C19—H19A | 0.9800 |
| C1—C2 | 1.561 (3) | C19—H19B | 0.9800 |
| C1—H1 | 1.0000 | C19—H19C | 0.9800 |
| C2—C18 | 1.480 (3) | C20—C21 | 1.496 (3) |
| C2—C17 | 1.481 (3) | C21—C22 | 1.384 (3) |
| C2—C3 | 1.589 (3) | C21—C26 | 1.392 (3) |
| C3—C19 | 1.517 (3) | C22—C23 | 1.390 (4) |
| C3—C4 | 1.567 (3) | C22—H22 | 0.9500 |
| C4—C20 | 1.531 (3) | C23—C24 | 1.374 (6) |
| C4—C5 | 1.540 (3) | C23—H23 | 0.9500 |
| C4—H4 | 1.0000 | C24—C25 | 1.370 (6) |
| C5—C27 | 1.521 (3) | C24—H24 | 0.9500 |
| C5—C6 | 1.537 (3) | C25—C26 | 1.393 (4) |

| | | | |
|---|---|---|---|
| C5—H5 | 1.0000 | C25—H25 | 0.9500 |
| C6—H6A | 0.9900 | C26—H26 | 0.9500 |
| C6—H6B | 0.9900 | C27—C28 | 1.389 (3) |
| C7—C8 | 1.542 (3) | C27—C32 | 1.396 (3) |
| C7—C10 | 1.543 (3) | C28—C29 | 1.398 (3) |
| C7—H7 | 1.0000 | C28—H28 | 0.9500 |
| C8—C9 | 1.490 (3) | C29—C30 | 1.386 (3) |
| C9—H9A | 0.9800 | C29—H29 | 0.9500 |
| C9—H9B | 0.9800 | C30—C31 | 1.383 (3) |
| C9—H9C | 0.9800 | C30—H30 | 0.9500 |
| C10—C11 | 1.497 (3) | C31—C32 | 1.392 (3) |
| C11—C12 | 1.387 (3) | C31—H31 | 0.9500 |
| C11—C16 | 1.401 (3) | C32—H32 | 0.9500 |
| C12—C13 | 1.390 (3) | | |

| | | | |
|---|---|---|---|
| C3—O3—H3 | 108 (2) | C13—C12—H12 | 120.0 |
| C6—C1—C7 | 112.72 (15) | C14—C13—C12 | 120.5 (2) |
| C6—C1—C2 | 108.66 (15) | C14—C13—H13 | 119.8 |
| C7—C1—C2 | 111.08 (15) | C12—C13—H13 | 119.8 |
| C6—C1—H1 | 108.1 | C15—C14—C13 | 119.7 (2) |
| C7—C1—H1 | 108.1 | C15—C14—H14 | 120.2 |
| C2—C1—H1 | 108.1 | C13—C14—H14 | 120.2 |
| C18—C2—C17 | 106.12 (16) | C16—C15—C14 | 120.2 (2) |
| C18—C2—C1 | 110.26 (16) | C16—C15—H15 | 119.9 |
| C17—C2—C1 | 111.54 (16) | C14—C15—H15 | 119.9 |
| C18—C2—C3 | 108.05 (15) | C15—C16—C11 | 120.4 (2) |
| C17—C2—C3 | 109.92 (16) | C15—C16—H16 | 119.8 |
| C1—C2—C3 | 110.78 (15) | C11—C16—H16 | 119.8 |
| O3—C3—C19 | 111.25 (17) | N1—C17—C2 | 178.2 (2) |
| O3—C3—C4 | 110.00 (16) | N2—C18—C2 | 178.2 (2) |
| C19—C3—C4 | 112.97 (17) | C3—C19—H19A | 109.5 |
| O3—C3—C2 | 104.92 (15) | C3—C19—H19B | 109.5 |
| C19—C3—C2 | 110.11 (16) | H19A—C19—H19B | 109.5 |
| C4—C3—C2 | 107.20 (15) | C3—C19—H19C | 109.5 |
| C20—C4—C5 | 110.67 (15) | H19A—C19—H19C | 109.5 |
| C20—C4—C3 | 108.84 (15) | H19B—C19—H19C | 109.5 |
| C5—C4—C3 | 110.81 (15) | O4—C20—C21 | 121.07 (18) |
| C20—C4—H4 | 108.8 | O4—C20—C4 | 120.11 (18) |
| C5—C4—H4 | 108.8 | C21—C20—C4 | 118.68 (17) |
| C3—C4—H4 | 108.8 | C22—C21—C26 | 119.3 (2) |
| C27—C5—C6 | 108.92 (15) | C22—C21—C20 | 122.9 (2) |
| C27—C5—C4 | 112.99 (15) | C26—C21—C20 | 117.7 (2) |
| C6—C5—C4 | 109.95 (15) | C21—C22—C23 | 120.4 (3) |
| C27—C5—H5 | 108.3 | C21—C22—H22 | 119.8 |
| C6—C5—H5 | 108.3 | C23—C22—H22 | 119.8 |
| C4—C5—H5 | 108.3 | C24—C23—C22 | 119.8 (3) |
| C1—C6—C5 | 112.69 (15) | C24—C23—H23 | 120.1 |
| C1—C6—H6A | 109.1 | C22—C23—H23 | 120.1 |

| | | | |
|---|---|---|---|
| C5—C6—H6A | 109.1 | C25—C24—C23 | 120.5 (3) |
| C1—C6—H6B | 109.1 | C25—C24—H24 | 119.7 |
| C5—C6—H6B | 109.1 | C23—C24—H24 | 119.7 |
| H6A—C6—H6B | 107.8 | C24—C25—C26 | 120.1 (3) |
| C8—C7—C10 | 106.82 (15) | C24—C25—H25 | 119.9 |
| C8—C7—C1 | 109.36 (15) | C26—C25—H25 | 119.9 |
| C10—C7—C1 | 111.73 (15) | C21—C26—C25 | 119.8 (3) |
| C8—C7—H7 | 109.6 | C21—C26—H26 | 120.1 |
| C10—C7—H7 | 109.6 | C25—C26—H26 | 120.1 |
| C1—C7—H7 | 109.6 | C28—C27—C32 | 118.43 (18) |
| O1—C8—C9 | 122.77 (18) | C28—C27—C5 | 121.58 (17) |
| O1—C8—C7 | 119.93 (18) | C32—C27—C5 | 119.82 (18) |
| C9—C8—C7 | 117.30 (17) | C27—C28—C29 | 120.97 (18) |
| C8—C9—H9A | 109.5 | C27—C28—H28 | 119.5 |
| C8—C9—H9B | 109.5 | C29—C28—H28 | 119.5 |
| H9A—C9—H9B | 109.5 | C30—C29—C28 | 119.9 (2) |
| C8—C9—H9C | 109.5 | C30—C29—H29 | 120.0 |
| H9A—C9—H9C | 109.5 | C28—C29—H29 | 120.0 |
| H9B—C9—H9C | 109.5 | C31—C30—C29 | 119.51 (19) |
| O2—C10—C11 | 121.19 (17) | C31—C30—H30 | 120.2 |
| O2—C10—C7 | 119.91 (17) | C29—C30—H30 | 120.2 |
| C11—C10—C7 | 118.70 (16) | C30—C31—C32 | 120.6 (2) |
| C12—C11—C16 | 119.29 (18) | C30—C31—H31 | 119.7 |
| C12—C11—C10 | 123.02 (17) | C32—C31—H31 | 119.7 |
| C16—C11—C10 | 117.66 (18) | C31—C32—C27 | 120.5 (2) |
| C11—C12—C13 | 119.9 (2) | C31—C32—H32 | 119.7 |
| C11—C12—H12 | 120.0 | C27—C32—H32 | 119.7 |

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| C6—C1—C2—C18 | 178.10 (16) | O2—C10—C11—C12 | 179.81 (19) |
| C7—C1—C2—C18 | −57.3 (2) | C7—C10—C11—C12 | 4.9 (3) |
| C6—C1—C2—C17 | −64.26 (19) | O2—C10—C11—C16 | 1.6 (3) |
| C7—C1—C2—C17 | 60.3 (2) | C7—C10—C11—C16 | −173.24 (18) |
| C6—C1—C2—C3 | 58.5 (2) | C16—C11—C12—C13 | −0.1 (3) |
| C7—C1—C2—C3 | −176.92 (15) | C10—C11—C12—C13 | −178.23 (19) |
| C18—C2—C3—O3 | −63.67 (19) | C11—C12—C13—C14 | −0.6 (4) |
| C17—C2—C3—O3 | −179.06 (15) | C12—C13—C14—C15 | 0.6 (4) |
| C1—C2—C3—O3 | 57.22 (19) | C13—C14—C15—C16 | −0.1 (4) |
| C18—C2—C3—C19 | 56.1 (2) | C14—C15—C16—C11 | −0.6 (4) |
| C17—C2—C3—C19 | −59.3 (2) | C12—C11—C16—C15 | 0.6 (3) |
| C1—C2—C3—C19 | 177.02 (17) | C10—C11—C16—C15 | 178.9 (2) |
| C18—C2—C3—C4 | 179.40 (16) | C5—C4—C20—O4 | 34.1 (2) |
| C17—C2—C3—C4 | 64.01 (19) | C3—C4—C20—O4 | −87.9 (2) |
| C1—C2—C3—C4 | −59.7 (2) | C5—C4—C20—C21 | −150.15 (17) |
| O3—C3—C4—C20 | 67.89 (19) | C3—C4—C20—C21 | 87.8 (2) |
| C19—C3—C4—C20 | −57.1 (2) | O4—C20—C21—C22 | −155.3 (2) |
| C2—C3—C4—C20 | −178.56 (15) | C4—C20—C21—C22 | 29.0 (3) |
| O3—C3—C4—C5 | −54.0 (2) | O4—C20—C21—C26 | 27.6 (3) |
| C19—C3—C4—C5 | −179.00 (17) | C4—C20—C21—C26 | −148.07 (19) |

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| C2—C3—C4—C5 | 59.5 (2) | C26—C21—C22—C23 | 0.7 (3) |
| C20—C4—C5—C27 | 58.4 (2) | C20—C21—C22—C23 | −176.3 (2) |
| C3—C4—C5—C27 | 179.24 (15) | C21—C22—C23—C24 | 1.7 (4) |
| C20—C4—C5—C6 | −179.68 (15) | C22—C23—C24—C25 | −1.7 (4) |
| C3—C4—C5—C6 | −58.8 (2) | C23—C24—C25—C26 | −0.6 (4) |
| C7—C1—C6—C5 | 179.09 (16) | C22—C21—C26—C25 | −2.9 (3) |
| C2—C1—C6—C5 | −57.3 (2) | C20—C21—C26—C25 | 174.2 (2) |
| C27—C5—C6—C1 | −177.85 (16) | C24—C25—C26—C21 | 2.9 (4) |
| C4—C5—C6—C1 | 57.8 (2) | C6—C5—C27—C28 | −70.2 (2) |
| C6—C1—C7—C8 | −35.8 (2) | C4—C5—C27—C28 | 52.3 (2) |
| C2—C1—C7—C8 | −158.06 (16) | C6—C5—C27—C32 | 105.0 (2) |
| C6—C1—C7—C10 | −153.88 (16) | C4—C5—C27—C32 | −132.47 (19) |
| C2—C1—C7—C10 | 83.90 (19) | C32—C27—C28—C29 | 0.0 (3) |
| C10—C7—C8—O1 | −132.18 (18) | C5—C27—C28—C29 | 175.32 (18) |
| C1—C7—C8—O1 | 106.7 (2) | C27—C28—C29—C30 | −1.0 (3) |
| C10—C7—C8—C9 | 47.5 (2) | C28—C29—C30—C31 | 1.1 (3) |
| C1—C7—C8—C9 | −73.6 (2) | C29—C30—C31—C32 | −0.1 (3) |
| C8—C7—C10—O2 | −91.6 (2) | C30—C31—C32—C27 | −0.9 (3) |
| C1—C7—C10—O2 | 27.9 (2) | C28—C27—C32—C31 | 0.9 (3) |
| C8—C7—C10—C11 | 83.3 (2) | C5—C27—C32—C31 | −174.44 (19) |
| C1—C7—C10—C11 | −157.13 (16) | | |

*Hydrogen-bond geometry (Å, º)*

*Cg*4 is a centroid of the C27–C32 phenyl ring.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O3—H3···O1$^{i}$ | 0.94 (3) | 1.89 (3) | 2.787 (2) | 159 (3) |
| C1—H1···O2 | 1.00 | 2.38 | 2.815 (2) | 106 |
| C1—H1···O3 | 1.00 | 2.48 | 2.855 (2) | 102 |
| C1—H1···N1$^{i}$ | 1.00 | 2.50 | 3.466 (3) | 163 |
| C5—H5···O3 | 1.00 | 2.53 | 2.892 (2) | 101 |
| C12—H12···O4$^{ii}$ | 0.95 | 2.58 | 3.242 (3) | 127 |
| C28—H28···O2$^{ii}$ | 0.95 | 2.40 | 3.319 (2) | 164 |
| C14—H14···*Cg*4$^{iii}$ | 0.95 | 2.80 | 3.475 (2) | 129 |

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