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Crystal structure and Hirshfeld surface analysis of (2,7-dihydroxynaphthalen-1-yl)(4-methylphenyl)-methanone

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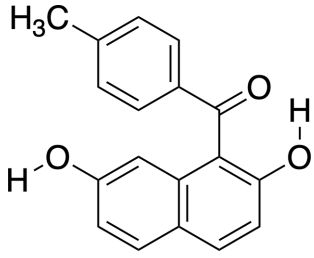
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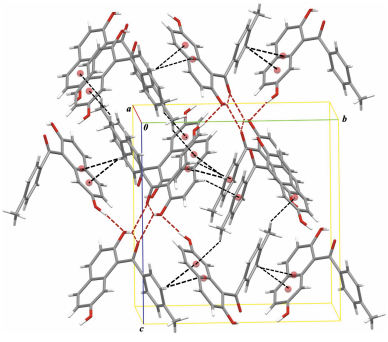
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In the title compound, $C_{18}H_{14}O_3$, an intramolecular O—H···O=C hydrogen bond is formed between the hydroxy and carbonyl groups on the naphthalene ring system, resulting in an $S(6)$ ring. An intramolecular C—H···π interaction between the C—H group at the 8-position of the naphthalene ring and the 4-methylphenyl ring is also observed. The torsion angles between the C=O group and the 4-methylphenyl and naphthalene rings are 142.58 (13) and 149.32 (12)°, respectively, and the dihedral angle between the two aromatic rings is 58.98 (5)°. In the crystal, two molecules are connected *via* a pair of intermolecular O—H···O=C hydrogen bonds between the carbonyl and 2-hydroxy groups, forming a centrosymmetric dimer with an $R_2^2(4)$ graph-set motif. These dimers are further linked into tetrameric aggregates through O—H···O hydrogen bonds between the 7-hydroxy and 2-hydroxy groups, and bifurcated C—H···π interactions. These tetramers are subsequently arranged into a crystal packing network *via* additional C—H···π interactions.

1. Chemical context

The design and control of crystal architectures *via* non-covalent interactions remain a central theme in crystal engineering and supramolecular chemistry.





Traditionally, the construction of predictable crystal structures has relied on the arrangement of robust supramolecular synthons sustained by classical strong hydrogen bonds (Desiraju, 1995). However, to precisely engineer and tailor desired crystalline architectures, a comprehensive understanding of the cooperativity and synergy among diverse weak interactions has become indispensable (Roman *et al.*, 2010).

Among these, C—H···π interactions play a pivotal role in determining molecular orientation and packing motifs in organic crystals. Generally, the investigation of weak interactions can be challenging in planar aromatic systems due to the dominance of strong parallel π–π stacking (Nishio *et al.*, 2014; Hunter *et al.*, 1990). To overcome this limitation, designing molecular systems with non-coplanar assembled aromatic rings offers an effective strategy; the steric hindrance

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

*Cg*2 is the centroid of the C5–C10 ring, *Cg*3 of the C12–C17 ring and *Cg*4 of the C1–C4/C10/C5–C9 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O1	0.97	1.68	2.5708 (1)	149
O2—H2···O1^{i}	0.97	2.53	3.1043 (1)	118
O3—H3···O2^{ii}	0.91	1.88	2.7800 (1)	166
C8—H8···*Cg*3	0.95	2.97	3.7209 (1)	137
C4—H4···*Cg*3^{iii}	0.95	2.84	3.5610 (1)	134
C16—H16···*Cg*2^{iv}	0.95	2.85	3.3853 (1)	117
C16—H16···*Cg*4^{iv}	0.95	2.97	3.6257 (1)	127
C18—H18*B*···*Cg*2^{v}	0.98	2.85	3.7250 (1)	149

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

inherently suppresses traditional π–π stacking, providing an ideal platform to clearly observe and analyze weaker, C—H-mediated interactions. When strong hydrogen-bonding components are intentionally integrated into such non-coplanar platforms, it becomes possible to systematically investigate the interplay, competition or cooperativity between strong and weak non-covalent networks.

In this context, the authors have extensively investigated the formation reactions and spatial structures of compounds bearing sterically congested non-coplanar aromatic rings (Okamoto & Yonezawa, 2009; Okamoto *et al.*, 2011). Notably, *peri*-aroylnaphthalene compounds and their homologues usually exhibit an excellent tendency toward single crystallinity, and the crystal structures of over 100 homologues and related compounds have been successfully determined by our group (Okamoto *et al.*, 2015). By systematically comparing the molecular conformations and crystal packing across these homologous series, the authors have clarified the relationships between molecular structure and intermolecular interactions. This accumulated body of work strongly suggests that weak interactions involving C—H hydrogens play critical roles in determining the crystalline spatial placement of molecules (Iida *et al.*, 2023; Li *et al.*, 2024; Li *et al.*, 2025).

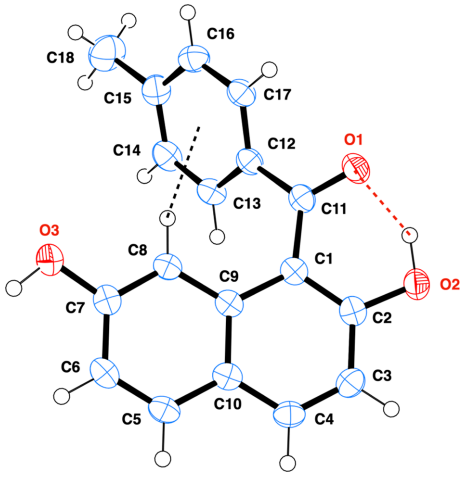


Figure 1
The molecular structure of the title compound, showing 50% probability displacement ellipsoids. Two types of intramolecular interactions are shown as dashed lines.

Herein, as part of our ongoing research into these non-coplanar aromatic ring systems, we report the crystal structure and Hirshfeld surface analysis of the title 1-(4-methylbenzoyl)naphthalene compound, namely, (2,7-dihydroxynaphthalen-1-yl)(4-methylphenyl)methanone.

2. Structural commentary

The molecular structure of the title molecule is shown in Fig. 1. Two types of intramolecular interactions are observed. An intramolecular O—H···O=C hydrogen bond is formed between the hydroxy and carbonyl groups on the naphthalene ring system (O2—H2···O1, 1.68 Å; Table 1), resulting in an *S*(6) ring (Etter *et al.*, 1990; Bernstein *et al.*, 1995). In addition, an intramolecular C—H···π interaction is observed between the H atom at the 8-position of the naphthalene ring and the benzene ring (C8—H8···*Cg*3, 2.97 Å). The benzene and naphthalene rings are tilted to a similar extent relative to the central carbonyl moiety [torsion angles: O1—C11—C12—C13 = 142.58 (13) and C9—C1—C11—O1 = 149.32 (12)°. The dihedral angle between the benzene and naphthalene rings is 58.98 (5)°.

3. Supramolecular features

In the crystal, two molecules are connected *via* pairs of intermolecular O—H···O=C hydrogen bonds between the carbonyl group and adjacent hydroxy group (2-hydroxy group) (O2—H2···O1, 2.53 Å; Table 1 and Fig. 2), forming centrosymmetric dimers with an $R_2^2(4)$ graph-set motif (Etter *et al.*, 1990; Bernstein *et al.*, 1995). The two hydroxy groups involved in the four-membered H···O···H···O hydrogen-bonded ring of the centrosymmetric dimer each act as hy-

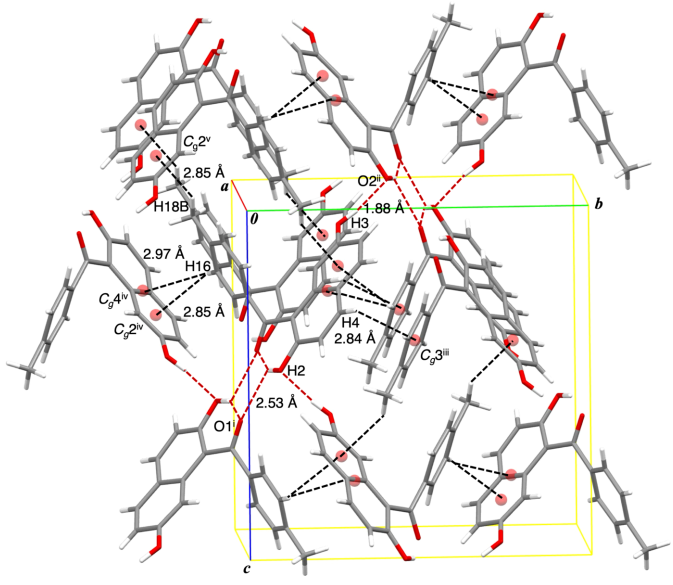


Figure 2
The crystal packing of the title compound, viewed down the *a* axis, illustrating the intermolecular O—H···O hydrogen-bonding interactions and C—H···π interactions, shown as red dashed lines and black dashed lines, respectively.

drogen-bond acceptors to form O—H···O hydrogen bonds with neighbouring molecules (O3—H3···O2, 1.88 Å). These four molecules are further stabilized *via* bifurcated C—H···π interactions (C16—H16···*Cg*2, 2.85 Å; C16—H16···*Cg*4, 2.97 Å). Furthermore, this tetrameric assembly is linked by two types of C—H···π interactions, forming a three-dimensional supramolecular network (C4—H4···*Cg*3, 2.84 Å; C18—H18*B*···*Cg*2, 2.85 Å).

4. Database survey

A search of the Cambridge Structural Database (CSD, Version 6.00, update August 2025; Groom *et al.*, 2016) found 22 instances of molecules involving the 1-benzoylnaphthalene skeleton, most of which possessed substituents at the 8-, 2,7- or 3,6-positions of the naphthalene ring core (CSD refcode LESLUS: Sakamoto *et al.*, 2013; VABLAT: Hijikata *et al.*, 2010; PUKGIM: Mitsui *et al.*, 2010; SOLXIB: Mitsui *et al.*, 2009; LANWEE: Sakthivel *et al.*, 2012; BOMRIG: Mohri *et al.*, 2014; AXAMES: Muto *et al.*, 2011). Among them, five compounds possessed a hydroxy group at the 2-position (OBOGEW: Li, 2026; VOJFOQ: Mitsui *et al.*, 2008; UCUHAE: Okamoto *et al.*, 2014; KABJUU: Nagasawa, Mitsui, Kato *et al.*, 2010; VABBEH: Nagasawa, Mitsui, Okamoto & Yonezawa, 2010). On the other hand, while two examples of 1,8-dibenzoylnaphthalene compounds with hydroxy groups at the 2,7-positions have been reported (LESLOM: Hijikata *et al.*, 2013; CIQBUB: Mohri *et al.*, 2013), no such reports exist for the corresponding 1-benzoylnaphthalene derivatives.

The title compound is compared with its 7-methoxy homologue, (2-hydroxy-7-methoxynaphthalen-1-yl)(4-methylphenyl)methanone (VABBEH), in which a methoxy group replaces the 7-hydroxy group. Both compounds have an intramolecular hydrogen bond between the carbonyl and 2-hydroxy groups, forming a six-membered O—H···O ring. In contrast to the 7-methoxy homologue, the naphthalene and 4-methylphenyl rings in the title compound are arranged with a more pronounced twist relative to the carbonyl moiety [the torsion angles between the C=O moiety and the two aromatic rings (benzene and the naphthalene rings): 142.58 (13) and 149.32 (12)° for the title compound, and 128.33 (13) and 158.84 (12)° for the 7-methoxy homologue].

In the crystal structures, both compounds form centrosymmetric dimers linked by intermolecular O—H···O=C hydrogen bonds between the hydroxy and carbonyl groups. The H···O distance of this intermolecular O—H···O=C hydrogen bond is shorter in the 7-methoxy homologue than in the title compound [2.33 *versus* 2.53 Å]. In the title compound, these centrosymmetric dimers are further connected into tetrameric aggregates *via* intermolecular O—H···O—H hydrogen bonds involving the 7-hydroxy group (1.88 Å) and bifurcated C—H···π interactions (2.85 and 2.97 Å); these tetramers are subsequently arranged through two types of C—H···π interactions (2.84 and 2.85 Å). In contrast, in the 7-methoxy homologue, the centrosymmetric dimers are directly linked into a three-dimensional packing network by three types of C—H···π interactions: (i) between a naphthalene C—H group and the benzene moiety of the naphthalene ring system (2.71 Å), (ii) between an aromatic C—H group of the 4-methylphenyl ring and the benzene moiety of the naphthalene system (2.76 Å), and (iii) between a methyl C—H group and the benzene ring of a 4-methylphenyl group (2.88 Å). Notably, these C—H···π interaction distances in the 7-methoxy homologue are generally shorter than those observed in the title compound.

5. Hirshfeld surface analysis

A Hirshfeld surface analysis was performed using *CrystalExplorer21* to examine and quantify the intermolecular contacts governing the crystal packing (Spackman *et al.*, 2021). The Hirshfeld surface of the title compound mapped over d_{norm} in the range −0.6253 to 1.3655 a.u. is shown in Fig. 3. On the d_{norm} surface, red, white and blue regions correspond to contacts shorter than, approximately equal to and longer than the sum of the van der Waals radii, respectively. Here, the two intense red spots result from the O3—H3···O2 hydrogen bonds (1.88 Å; Table 1). A pair of adjacent small red spots arises from a pair of O2—H2···O1 hydrogen bonds (2.53 Å; Table 1). The percentage contributions of the intermolecular

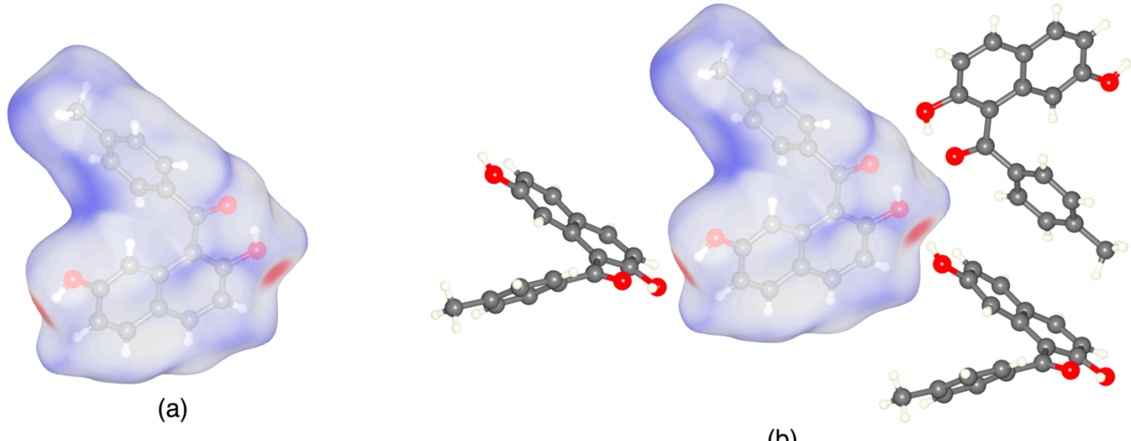


Figure 3
(*a*) The Hirshfeld surface of the title compound mapped over d_{norm}. (*b*) Several large red points are assigned as O1···H2 and O2···H3 short contacts.

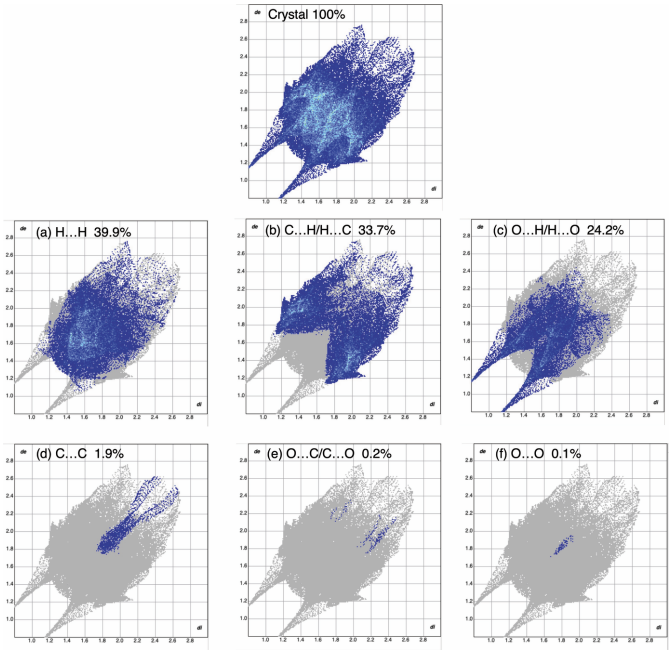


Figure 4
The full two-dimensional fingerprint plot for the title compound, and those delineated into (*a*) H···H, (*b*) C···H/H···C, (*c*) O···H/H···O, (*d*) C···C, (*e*) O···C/C···O and (*f*) O···O contacts.

interactions to the total Hirshfeld surface were computed by two-dimensional fingerprint plots. As illustrated in Fig. 4, the d_i *versus* d_e plots show that H···H contacts make the largest contribution (39.9%). The C···H/H···C contacts (33.7%) are present as a relatively large contributor. The O···H/H···O contacts contribute 24.2% to the total Hirshfeld surface and are characterized by a pair of prominent spikes in the two-dimensional fingerprint plot. These features are consistent with several kinds of C—H···π interactions and the O2—H2···O1 hydrogen bonding interaction observed in the crystal (Fig. 2 and Table 1). Other contacts contribute to a lesser extent, namely, C···C (1.9%), O···C/C···O (0.2%) and O···O (0.1%).

For comparison, a Hirshfeld surface analysis was also examined for the 7-methoxy homologue (VABBEH). Although both compounds form centrosymmetric dimers *via* intermolecular O—H···O═C hydrogen bonds (2.33 (2) Å in VABBEH), the influence of the dimeric motif on the overall molecular packing differs markedly from that in the title compound. On the d_{norm} surface of VABBEH (mapped over the range −0.2838 to 1.8299 a.u.), the largest red spots are located adjacent to each other in the vicinity of the carbonyl and hydroxy groups (Fig. 5, left), while several small red spots are observed around the naphthalene ring core and the 4-methylbenzoyl group. Collectively, these close contacts involve a total of seven neighbouring molecules around the central unit, a number significantly larger than the three observed for the title compound (Fig. 5 *versus* Fig. 3, right). In the two-dimensional fingerprint plots, H···H contacts are the major contributor (49.4%), while C···H/H···C contacts (28.1%) and O···H/H···O contacts (17.7%) make smaller contributions. Although the order of contribution from these three interactions remains unchanged compared to the title compound, the combined contribution of H···H and C···H/H···C contacts is notably larger relative to the O···H/H···O contacts. This feature further confirms that the O···H/H···O interactions in VABBEH are confined to the dimeric unit, whereas weaker interactions, such as C—H···π and van der Waals contacts, govern the three-dimensional crystal packing in the case of the 7-methoxy homologue.

6. Synthesis and crystallization

To a solution of 2,7-dihydroxynaphthalene (320 mg, 2.0 mmol) and 4-methylbenzoyl chloride (309 mg, 2.0 mmol) was added $TiCl_4$ (1.14 g, 6.0 mmol). The reaction mixture was stirred for

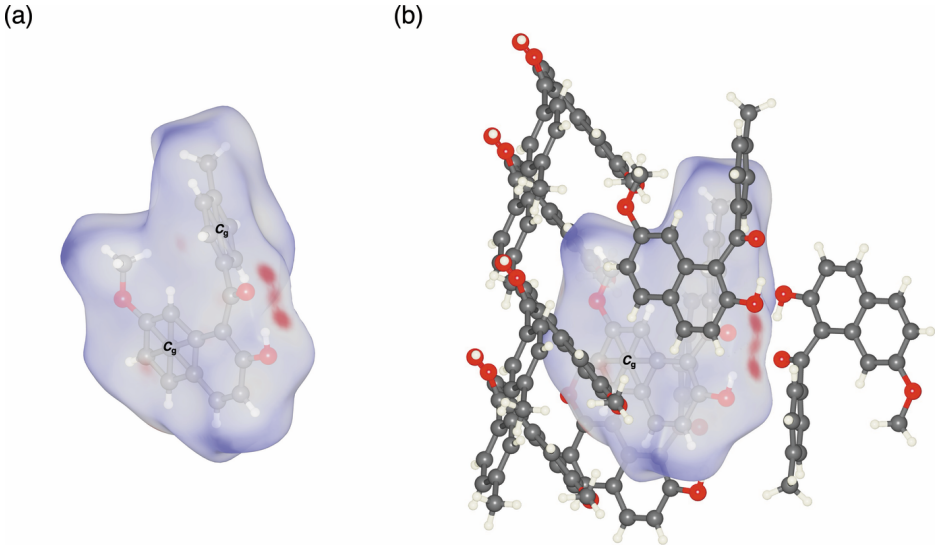


Figure 5
(*a*) The Hirshfeld surface of the 7-methoxy homologue mapped over d_{norm}. (*b*) Several close contacts involve a total of seven neighbouring molecules around the central unit.

3 h to give a dark-red solution, which was then poured into ice–water (30 ml). The aqueous layer was extracted with $CHCl_3$ (30 ml × 3). The combined organic layers were washed with saturated $NaHCO_3$(aq), followed by brine (30 ml × 3), and dried over $MgSO_4$ overnight. The solvent was removed *in vacuo*. The crude material was dissolved in hexane, heated on an oil bath for 15–20 min and then allowed to stand undisturbed for slow evaporation at room temperature to give the title compound as yellow platelets (yield: 239 mg, 43%; m.p. 439–440 K). ^{1}H NMR (300 MHz, $CDCl_3$) δ 11.14 (1H, *s*), 7.84 (1H, *d*, *J* = 9.0 Hz), 7.65 (1H, *d*, *J* = 8.4 Hz), 7.56 (2H, *d*, *J* = 8.1 Hz), 7.22 (2H, *d*, *J* = 8.1 Hz), 7.07 (1H, *d*, *J* = 9.0 Hz), 6.88 (1H, *dd*, *J* = 8.7 and 2.4 Hz), 6.66 (1H, *ds*, *J* = 2.4 Hz), 4.85 (1H, *s*), 2.43 (3H, *s*) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ 199.92, 161.77, 154.20, 143.68, 137.20, 135.92, 134.12, 130.53, 129.69, 129.29, 123.75, 116.51, 114.86, 109.70, 21.730 ppm. IR (KBr): ν = 3391 (O—H), 1632 (C═O), 1602 (Ar), 1582 (Ar) cm^{-1}. HRMS (FAB): *m*/*z* calculated for $C_{18}H_{15}O_3$ = 279.1016 [M + H]$^+$; found: 279.1055.

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The hydroxy H atoms (H2 and H3) were located from residual electron-density peaks in a difference Fourier map and refined freely with isotropic displacement parameters. The resulting O—H bond lengths were found to be O2—H2 = 0.97 (2) Å and O3—H3 = 0.91 (2) Å. The H atoms attached to C atoms were introduced in calculated positions and treated as riding on their parent atoms, with C—H = 0.98 (methyl) or 0.95 Å (aromatic), with U_{iso}(H) = 1.2U_{eq}(aromatic) or U_{iso}(H) = 1.5U_{eq}(methyl).

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{18}H_{14}O_3$
M_r	278.29
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	193
a, *b*, *c* (Å)	6.6476 (1), 14.2658 (3), 14.5476 (3)
β (°)	97.5163 (13)
V (Å^3)	1367.74 (5)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.74
Crystal size (mm)	0.50 × 0.25 × 0.10
Data collection	
Diffractometer	Rigaku R-AXIS RAPID
Absorption correction	Numerical (*NUMABS*; Rigaku, 1999)
T_{min}, T_{max}	0.712, 0.928
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	23674, 2494, 2162
R_{int}	0.052
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.602
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.038, 0.113, 1.10
No. of reflections	2494
No. of parameters	199
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.26, −0.21

Computer programs: *PROCESS-AUTO* (Rigaku, 1998), *SIR2004* (Burla *et al.*, 2005), *SHELXL2014* (Sheldrick, 2015) and *ORTEPIII* (Burnett & Johnson, 1996).

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Crystal structure and Hirshfeld surface analysis of (2,7-dihydroxynaphthalen-1-yl)(4-methylphenyl)methanone

Hiroaki Iitsuka, Kun Li, Jiali Yao, Keiichi Noguchi and Akiko Okamoto

Computing details

(2,7-Dihydroxynaphthalen-1-yl)(4-methylphenyl)methanone

Crystal data

$C_{18}H_{14}O_3$
M_r = 278.29
Monoclinic, $P2_1/n$
a = 6.6476 (1) Å
b = 14.2658 (3) Å
c = 14.5476 (3) Å
β = 97.5163 (13)°
V = 1367.74 (5) Å^3
Z = 4
$F(000)$ = 584
D_x = 1.351 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54187 Å
Cell parameters from 18243 reflections
θ = 3.1–68.3°
μ = 0.74 mm^{-1}
T = 193 K
Platelet, yellow
0.50 × 0.25 × 0.10 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Detector resolution: 10.000 pixels mm^{-1}
ω scans
Absorption correction: numerical (NUMABS; Rigaku, 1999)
T_{min} = 0.712, T_{max} = 0.928
23674 measured reflections
2494 independent reflections
2162 reflections with $I > 2\sigma(I)$
R_{int} = 0.052
θ_{max} = 68.3°, θ_{min} = 4.4°
h = −7→8
k = −17→17
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.113
S = 1.10
2494 reflections
199 parameters
0 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0669P)^2 + 0.1455P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.05916 (15)	0.02730 (7)	0.40390 (6)	0.0372 (3)
O3	0.19612 (15)	0.27544 (8)	0.05987 (7)	0.0415 (3)
O2	−0.29747 (16)	0.08074 (6)	0.43134 (6)	0.0347 (3)
C9	−0.13829 (19)	0.18908 (9)	0.22016 (8)	0.0277 (3)
C1	−0.13930 (19)	0.12412 (9)	0.29672 (8)	0.0283 (3)
C8	0.02779 (19)	0.19993 (9)	0.16972 (8)	0.0299 (3)
H8	0.1423	0.1600	0.1826	0.036*
C10	−0.3070 (2)	0.25068 (9)	0.19884 (9)	0.0296 (3)
C6	−0.1436 (2)	0.32582 (9)	0.07895 (9)	0.0332 (3)
H6	−0.1451	0.3708	0.0307	0.040*
C4	−0.4679 (2)	0.24842 (9)	0.25358 (9)	0.0322 (3)
H4	−0.5827	0.2876	0.2374	0.039*
C11	0.01716 (19)	0.05177 (9)	0.32176 (8)	0.0289 (3)
C12	0.1199 (2)	0.00263 (9)	0.25062 (8)	0.0289 (3)
C7	0.0263 (2)	0.26741 (9)	0.10224 (9)	0.0313 (3)
C3	−0.4620 (2)	0.19149 (9)	0.32878 (9)	0.0336 (3)
H3A	−0.5703	0.1920	0.3654	0.040*
C15	0.3254 (2)	−0.09102 (10)	0.12113 (10)	0.0372 (3)
C2	−0.2945 (2)	0.13176 (9)	0.35206 (8)	0.0297 (3)
C13	0.0207 (2)	−0.01846 (9)	0.16231 (9)	0.0333 (3)
H13	−0.1165	−0.0001	0.1455	0.040*
C5	−0.3056 (2)	0.31693 (9)	0.12665 (9)	0.0324 (3)
H5	−0.4205	0.3562	0.1111	0.039*
C17	0.3201 (2)	−0.02630 (9)	0.27421 (9)	0.0318 (3)
H17	0.3872	−0.0152	0.3350	0.038*
C14	0.1219 (2)	−0.06598 (10)	0.09941 (9)	0.0374 (3)
H14	0.0517	−0.0819	0.0404	0.045*
C16	0.4215 (2)	−0.07086 (10)	0.20990 (10)	0.0369 (3)
H16	0.5595	−0.0882	0.2264	0.044*
C18	0.4392 (3)	−0.13897 (13)	0.05151 (12)	0.0581 (5)
H18A	0.5601	−0.1023	0.0432	0.087*
H18B	0.3514	−0.1440	−0.0079	0.087*
H18C	0.4800	−0.2018	0.0739	0.087*
H3	0.179 (3)	0.3261 (15)	0.0211 (14)	0.070 (6)*
H2	−0.163 (3)	0.0527 (13)	0.4423 (13)	0.063 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0451 (6)	0.0383 (6)	0.0281 (5)	0.0060 (4)	0.0039 (4)	0.0054 (4)
O3	0.0385 (6)	0.0470 (6)	0.0404 (6)	0.0014 (5)	0.0105 (4)	0.0140 (5)
O2	0.0423 (6)	0.0344 (5)	0.0284 (5)	0.0014 (4)	0.0089 (4)	0.0020 (4)
C9	0.0313 (7)	0.0234 (6)	0.0274 (6)	−0.0011 (5)	−0.0001 (5)	−0.0023 (5)
C1	0.0314 (7)	0.0250 (7)	0.0279 (6)	−0.0020 (5)	0.0016 (5)	−0.0022 (5)
C8	0.0307 (7)	0.0272 (7)	0.0310 (7)	0.0017 (5)	0.0015 (5)	0.0011 (5)

C10	0.0319 (7)	0.0256 (7)	0.0302 (7)	−0.0004 (5)	−0.0004 (5)	−0.0034 (5)
C6	0.0407 (8)	0.0282 (7)	0.0296 (6)	−0.0006 (6)	−0.0003 (5)	0.0034 (5)
C4	0.0305 (7)	0.0284 (7)	0.0369 (7)	0.0026 (5)	0.0009 (5)	−0.0039 (5)
C11	0.0319 (7)	0.0248 (7)	0.0295 (6)	−0.0040 (5)	0.0023 (5)	0.0016 (5)
C12	0.0340 (7)	0.0215 (6)	0.0307 (6)	−0.0003 (5)	0.0026 (5)	0.0026 (5)
C7	0.0344 (7)	0.0300 (7)	0.0291 (6)	−0.0037 (5)	0.0027 (5)	−0.0004 (5)
C3	0.0333 (7)	0.0333 (7)	0.0351 (7)	−0.0002 (6)	0.0082 (5)	−0.0043 (5)
C15	0.0461 (8)	0.0295 (7)	0.0372 (7)	0.0023 (6)	0.0094 (6)	−0.0025 (6)
C2	0.0359 (7)	0.0254 (7)	0.0277 (6)	−0.0033 (5)	0.0033 (5)	−0.0026 (5)
C13	0.0355 (7)	0.0297 (7)	0.0334 (7)	0.0027 (6)	−0.0005 (5)	0.0009 (5)
C5	0.0350 (7)	0.0264 (7)	0.0339 (7)	0.0023 (6)	−0.0024 (5)	−0.0003 (5)
C17	0.0364 (7)	0.0266 (7)	0.0316 (7)	0.0011 (5)	0.0011 (5)	0.0011 (5)
C14	0.0486 (8)	0.0317 (8)	0.0305 (7)	0.0016 (6)	−0.0001 (6)	−0.0028 (5)
C16	0.0344 (7)	0.0325 (7)	0.0437 (8)	0.0047 (6)	0.0046 (6)	0.0003 (6)
C18	0.0629 (11)	0.0611 (11)	0.0519 (10)	0.0097 (9)	0.0137 (8)	−0.0166 (8)

Geometric parameters (Å, º)

O1—C11	1.2411 (15)	C11—C12	1.4878 (18)
O3—C7	1.3601 (16)	C12—C17	1.3930 (19)
O3—H3	0.91 (2)	C12—C13	1.3978 (17)
O2—C2	1.3661 (15)	C3—C2	1.4081 (19)
O2—H2	0.97 (2)	C3—H3A	0.9500
C9—C8	1.4118 (18)	C15—C16	1.394 (2)
C9—C10	1.4266 (18)	C15—C14	1.395 (2)
C9—C1	1.4496 (17)	C15—C18	1.505 (2)
C1—C2	1.3932 (17)	C13—C14	1.3823 (19)
C1—C11	1.4768 (18)	C13—H13	0.9500
C8—C7	1.3741 (18)	C5—H5	0.9500
C8—H8	0.9500	C17—C16	1.3781 (19)
C10—C5	1.4137 (18)	C17—H17	0.9500
C10—C4	1.4151 (18)	C14—H14	0.9500
C6—C5	1.3614 (19)	C16—H16	0.9500
C6—C7	1.4088 (19)	C18—H18A	0.9800
C6—H6	0.9500	C18—H18B	0.9800
C4—C3	1.3589 (19)	C18—H18C	0.9800
C4—H4	0.9500		

C7—O3—H3	107.8 (12)	C4—C3—H3A	120.2
C2—O2—H2	104.0 (11)	C2—C3—H3A	120.2
C8—C9—C10	117.74 (11)	C16—C15—C14	118.07 (13)
C8—C9—C1	123.50 (11)	C16—C15—C18	120.49 (13)
C10—C9—C1	118.57 (12)	C14—C15—C18	121.44 (13)
C2—C1—C9	118.18 (11)	O2—C2—C1	122.67 (12)
C2—C1—C11	117.58 (11)	O2—C2—C3	115.28 (12)
C9—C1—C11	124.22 (11)	C1—C2—C3	122.03 (12)
C7—C8—C9	121.00 (12)	C14—C13—C12	120.15 (12)
C7—C8—H8	119.5	C14—C13—H13	119.9

C9—C8—H8	119.5	C12—C13—H13	119.9
C5—C10—C4	120.80 (12)	C6—C5—C10	121.55 (12)
C5—C10—C9	119.48 (12)	C6—C5—H5	119.2
C4—C10—C9	119.62 (12)	C10—C5—H5	119.2
C5—C6—C7	119.02 (12)	C16—C17—C12	120.40 (12)
C5—C6—H6	120.5	C16—C17—H17	119.8
C7—C6—H6	120.5	C12—C17—H17	119.8
C3—C4—C10	121.49 (12)	C13—C14—C15	121.14 (13)
C3—C4—H4	119.3	C13—C14—H14	119.4
C10—C4—H4	119.3	C15—C14—H14	119.4
O1—C11—C1	119.77 (11)	C17—C16—C15	121.23 (13)
O1—C11—C12	118.11 (11)	C17—C16—H16	119.4
C1—C11—C12	122.02 (10)	C15—C16—H16	119.4
C17—C12—C13	118.89 (12)	C15—C18—H18A	109.5
C17—C12—C11	118.61 (11)	C15—C18—H18B	109.5
C13—C12—C11	122.45 (12)	H18A—C18—H18B	109.5
O3—C7—C8	117.00 (12)	C15—C18—H18C	109.5
O3—C7—C6	121.87 (12)	H18A—C18—H18C	109.5
C8—C7—C6	121.13 (12)	H18B—C18—H18C	109.5
C4—C3—C2	119.60 (12)		

C8—C9—C1—C2	168.19 (11)	C5—C6—C7—O3	−177.23 (12)
C10—C9—C1—C2	−6.74 (17)	C5—C6—C7—C8	2.26 (19)
C8—C9—C1—C11	−9.98 (19)	C10—C4—C3—C2	−1.37 (19)
C10—C9—C1—C11	175.09 (11)	C9—C1—C2—O2	−172.94 (11)
C10—C9—C8—C7	−0.20 (18)	C11—C1—C2—O2	5.35 (17)
C1—C9—C8—C7	−175.17 (11)	C9—C1—C2—C3	8.46 (18)
C8—C9—C10—C5	2.46 (17)	C11—C1—C2—C3	−173.25 (11)
C1—C9—C10—C5	177.68 (11)	C4—C3—C2—O2	176.83 (11)
C8—C9—C10—C4	−173.90 (11)	C4—C3—C2—C1	−4.47 (19)
C1—C9—C10—C4	1.32 (17)	C17—C12—C13—C14	−1.0 (2)
C5—C10—C4—C3	−173.50 (12)	C11—C12—C13—C14	−178.65 (12)
C9—C10—C4—C3	2.81 (19)	C7—C6—C5—C10	0.08 (19)
C2—C1—C11—O1	−28.86 (17)	C4—C10—C5—C6	173.88 (12)
C9—C1—C11—O1	149.32 (12)	C9—C10—C5—C6	−2.44 (19)
C2—C1—C11—C12	147.68 (12)	C13—C12—C17—C16	3.2 (2)
C9—C1—C11—C12	−34.14 (18)	C11—C12—C17—C16	−179.08 (12)
O1—C11—C12—C17	−35.09 (18)	C12—C13—C14—C15	−2.2 (2)
C1—C11—C12—C17	148.32 (12)	C16—C15—C14—C13	3.1 (2)
O1—C11—C12—C13	142.58 (13)	C18—C15—C14—C13	−177.25 (14)
C1—C11—C12—C13	−34.01 (18)	C12—C17—C16—C15	−2.2 (2)
C9—C8—C7—O3	177.32 (11)	C14—C15—C16—C17	−0.9 (2)
C9—C8—C7—C6	−2.18 (19)	C18—C15—C16—C17	179.45 (14)

Hydrogen-bond geometry (Å, º)

*Cg*2 is the centroid of the C5–C10 ring, *Cg*3 of the C12–C17 ring and *Cg*4 of the C1–C4/C10/C5–C9 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O1	0.97	1.68	2.5708 (1)	149
O2—H2···O1^{i}	0.97	2.53	3.1043 (1)	118
O3—H3···O2^{ii}	0.91	1.88	2.7800 (1)	166
C8—H8···*Cg*3	0.95	2.97	3.7209 (1)	137
C4—H4···*Cg*3^{iii}	0.95	2.84	3.5610 (1)	134
C16—H16···*Cg*2^{iv}	0.95	2.85	3.3853 (1)	117
C16—H16···*Cg*4^{iv}	0.95	2.97	3.6257 (1)	127
C18—H18*B*···*Cg*2^{v}	0.98	2.85	3.7250 (1)	149

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