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Redetermined structure of 4-(benzyloxy)benzoic acid

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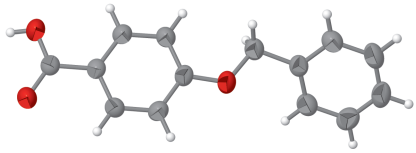
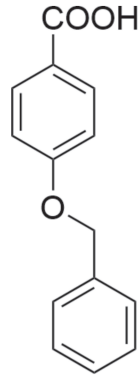
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{14}H_{14}O_3$, the dihedral angle between the aromatic rings is 39.76 (9)°. In the crystal, the molecules associate to form centrosymmetric acid–acid dimers linked by pairwise O—H···O hydrogen bonds. The precision of the geometric parameters in the present single-crystal study is about an order of magnitude better than the previous powder diffraction study [Chattopadhyay *et al.* (2013). *CrystEngComm*, **15**, 1077–1085].

3D view



Chemical scheme



Structure description

Non-steroidal anti-inflammatory drugs (NSAIDs) constitute approximately 5–10% of all prescribed medicines worldwide as antipyretic, anti-inflammatory and analgesic agents (Sohail *et al.*, 2023). Moreover, they are found to have a protective role against various critical diseases, such as cancer and cardiovascular diseases (Prasher & Sharma, 2021). It is estimated that 30 million individuals use NSAIDs daily (Bhala *et al.*, 2013).

As part of our studies in this area, the title compound (Fig. 1) was synthesized by a two-step reaction. The C1–C6 and C9–C14 aromatic rings subtend a dihedral angle of 39.76 (9)° and the linking C4—O3—C8—C9 bond has an *anti* conformation [torsion angle = −171.59 (12)°]. The short C4—O3 bond length of 1.3601 (16) Å indicates some conjugation of the O atom lone pair with the adjacent aromatic ring.

In the extended structure, molecules pair up to form carboxylic acid–carboxylic acid hydrogen-bonded dimers (Table 1, Fig. 2). The crystal structure of the title compound was also solved from X-ray powder diffraction data (Chattopadhyay *et al.*, 2013) with corresponding hydrogen-bond parameters of 1.94 Å and 176°, respectively, which deviate from those obtained in this study from single-crystal X-ray diffraction.

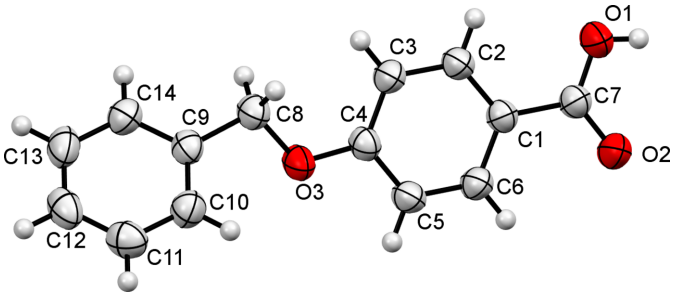


Figure 1
The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level.

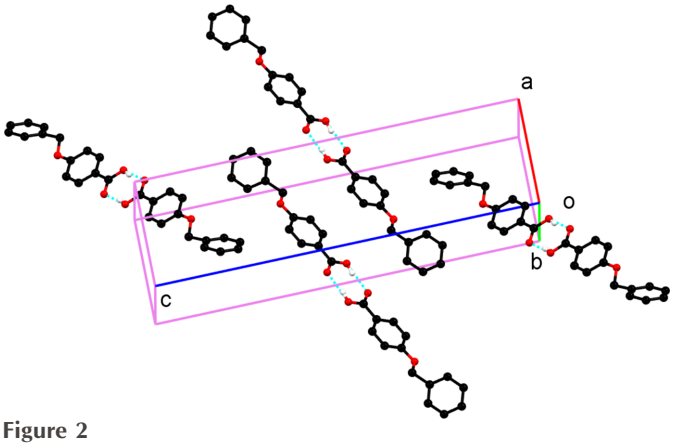


Figure 2
Packing of the molecules in the title compound (for clarity, H atoms not involved in hydrogen bonding are omitted).

Synthesis and crystallization

The title compound was prepared by a two-step reaction (Fig. 3). The product was purified by column chromatography. Single crystals were obtained by slowly evaporating an ethanol solution of the title compound.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

QS thanks the Graduate Innovation Fund of WIT for financial support.

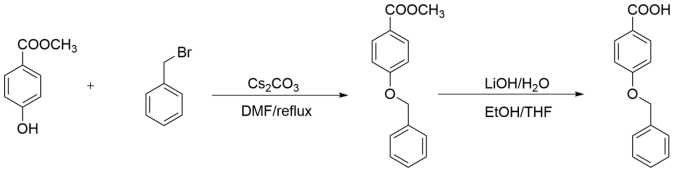


Figure 3
Synthesis of the title compound.

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2^{i}	0.82	1.81	2.6213 (15)	169

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{14}H_{12}O_3$
M_r	228.24
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	305
a, b, c (Å)	10.0564 (5), 3.9985 (2), 28.2235 (14)
β (°)	97.744 (5)
V (Å^3)	1124.54 (10)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.10
Crystal size (mm)	0.16 × 0.15 × 0.13
Data collection	
Diffractometer	XtaLAB Synergy R, DW system, HyPix
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2022)
T_{min}, T_{max}	0.875, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10523, 2793, 1996
R_{int}	0.019
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.721
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.044, 0.135, 1.08
No. of reflections	2793
No. of parameters	156
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.26, −0.19

Computer programs: *CrysAlis PRO* (Rigaku OD, 2022), *SHELXT2018/3* (Sheldrick, 2015*b*), *SHELXL2018/3* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

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full crystallographic data

IUCrData (2024). **9**, x240752 [https://doi.org/10.1107/S2414314624007521]

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4-(Benzyloxy)benzoic acid

Crystal data

$C_{14}H_{12}O_3$
M_r = 228.24
Monoclinic, $P2_1/n$
a = 10.0564 (5) Å
b = 3.9985 (2) Å
c = 28.2235 (14) Å
β = 97.744 (5)°
V = 1124.54 (10) Å^3
Z = 4
$F(000)$ = 480
D_x = 1.348 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5298 reflections
θ = 2.1–29.8°
μ = 0.10 mm^{-1}
T = 305 K
Block, clear light colourless
0.16 × 0.15 × 0.13 mm

Data collection

XtaLAB Synergy R, DW system, HyPix diffractometer
Radiation source: Rotating-anode X-ray tube, Rigaku (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.0000 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2022)
T_{min} = 0.875, T_{max} = 1.000
10523 measured reflections
2793 independent reflections
1996 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 30.8°, θ_{min} = 2.1°
h = −12→13
k = −4→5
l = −29→37

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.135
S = 1.08
2793 reflections
156 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0641P)^2 + 0.1717P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Extinction correction: *SHELXL2018/3* (Sheldrick, 2015b), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.008 (2)

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.

Refinement. The positions of the H atom attached to O1 was obtained from a difference Fourier map. Other H atoms were positioned geometrically with C—H = 0.93 Å and constrained to ride on their parent atoms with U_{iso}(H) = 1.2U_{eq}(C,*O*).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O3	0.35692 (9)	0.6152 (3)	0.36054 (4)	0.0557 (3)
O2	0.94382 (10)	0.2255 (4)	0.45018 (4)	0.0666 (4)
O1	0.83138 (10)	0.0288 (4)	0.50696 (4)	0.0658 (4)
H1	0.906153	−0.043118	0.517133	0.099*
C4	0.46870 (13)	0.5035 (4)	0.38859 (5)	0.0444 (3)
C5	0.58913 (14)	0.5572 (4)	0.37085 (5)	0.0503 (4)
H5	0.589311	0.663292	0.341539	0.060*
C6	0.70792 (13)	0.4545 (4)	0.39638 (5)	0.0491 (4)
H6	0.788198	0.490876	0.384244	0.059*
C1	0.70890 (12)	0.2960 (4)	0.44041 (4)	0.0431 (3)
C2	0.58902 (13)	0.2464 (4)	0.45795 (5)	0.0495 (4)
H2	0.589103	0.142972	0.487473	0.059*
C3	0.46833 (14)	0.3477 (4)	0.43243 (5)	0.0505 (4)
H3	0.388052	0.311700	0.444589	0.061*
C8	0.22907 (13)	0.5294 (4)	0.37376 (5)	0.0526 (4)
H8A	0.216021	0.642645	0.403176	0.063*
H8B	0.224207	0.290205	0.378971	0.063*
C9	0.12226 (13)	0.6340 (3)	0.33413 (5)	0.0452 (3)
C10	0.13472 (15)	0.5661 (4)	0.28683 (5)	0.0548 (4)
H10	0.211545	0.460996	0.279204	0.066*
C11	0.03322 (16)	0.6542 (4)	0.25081 (6)	0.0616 (4)
H11	0.042281	0.608856	0.219082	0.074*
C12	−0.08060 (15)	0.8081 (4)	0.26183 (6)	0.0625 (4)
H12	−0.148833	0.865897	0.237618	0.075*
C13	−0.09369 (15)	0.8766 (4)	0.30845 (6)	0.0623 (4)
H13	−0.170991	0.980303	0.315929	0.075*
C14	0.00810 (14)	0.7917 (4)	0.34459 (6)	0.0536 (4)
H14	−0.000845	0.841711	0.376181	0.064*
C7	0.83567 (13)	0.1772 (4)	0.46735 (5)	0.0467 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O3	0.0376 (5)	0.0723 (7)	0.0548 (6)	−0.0030 (5)	−0.0018 (4)	0.0171 (5)
O2	0.0403 (5)	0.1045 (10)	0.0549 (6)	0.0051 (6)	0.0064 (4)	0.0152 (6)
O1	0.0449 (6)	0.0978 (9)	0.0527 (6)	0.0062 (6)	0.0000 (5)	0.0210 (6)
C4	0.0394 (7)	0.0492 (8)	0.0425 (7)	−0.0025 (6)	−0.0015 (5)	0.0019 (6)
C5	0.0461 (7)	0.0633 (9)	0.0407 (7)	−0.0034 (6)	0.0029 (6)	0.0094 (6)
C6	0.0378 (7)	0.0648 (9)	0.0447 (7)	−0.0040 (6)	0.0056 (5)	0.0046 (6)
C1	0.0390 (7)	0.0499 (8)	0.0392 (6)	−0.0014 (5)	0.0011 (5)	−0.0005 (5)
C2	0.0434 (7)	0.0629 (9)	0.0417 (7)	−0.0008 (6)	0.0038 (5)	0.0089 (6)

C3	0.0378 (7)	0.0644 (10)	0.0492 (8)	−0.0020 (6)	0.0058 (5)	0.0083 (7)
C8	0.0405 (7)	0.0618 (9)	0.0546 (8)	−0.0032 (6)	0.0025 (6)	0.0103 (7)
C9	0.0374 (6)	0.0455 (8)	0.0515 (8)	−0.0053 (5)	0.0019 (5)	0.0046 (6)
C10	0.0462 (8)	0.0608 (10)	0.0573 (8)	0.0017 (7)	0.0068 (6)	−0.0025 (7)
C11	0.0607 (9)	0.0722 (11)	0.0497 (8)	−0.0119 (8)	−0.0004 (7)	0.0001 (7)
C12	0.0449 (8)	0.0691 (11)	0.0689 (10)	−0.0100 (7)	−0.0094 (7)	0.0173 (8)
C13	0.0396 (7)	0.0666 (11)	0.0803 (11)	0.0039 (7)	0.0063 (7)	0.0117 (8)
C14	0.0438 (7)	0.0609 (9)	0.0568 (8)	−0.0024 (7)	0.0090 (6)	0.0022 (7)
C7	0.0406 (7)	0.0584 (9)	0.0403 (7)	−0.0004 (6)	0.0022 (5)	−0.0007 (6)

Geometric parameters (Å, º)

O3—C4	1.3601 (16)	C3—H3	0.9300
O3—C8	1.4279 (17)	C8—H8A	0.9700
O2—C7	1.2636 (17)	C8—H8B	0.9700
O1—H1	0.8200	C8—C9	1.5019 (19)
O1—C7	1.2716 (17)	C9—C10	1.384 (2)
C4—C5	1.3881 (19)	C9—C14	1.376 (2)
C4—C3	1.3857 (19)	C10—H10	0.9300
C5—H5	0.9300	C10—C11	1.386 (2)
C5—C6	1.3721 (19)	C11—H11	0.9300
C6—H6	0.9300	C11—C12	1.372 (2)
C6—C1	1.3937 (19)	C12—H12	0.9300
C1—C2	1.3780 (18)	C12—C13	1.368 (2)
C1—C7	1.4723 (18)	C13—H13	0.9300
C2—H2	0.9300	C13—C14	1.386 (2)
C2—C3	1.3858 (19)	C14—H14	0.9300
C4—O3—C8	118.15 (11)	C9—C8—H8A	110.0
C7—O1—H1	109.5	C9—C8—H8B	110.0
O3—C4—C5	115.61 (12)	C10—C9—C8	121.00 (13)
O3—C4—C3	124.46 (12)	C14—C9—C8	120.09 (13)
C3—C4—C5	119.93 (12)	C14—C9—C10	118.89 (13)
C4—C5—H5	119.9	C9—C10—H10	119.9
C6—C5—C4	120.30 (12)	C9—C10—C11	120.29 (14)
C6—C5—H5	119.9	C11—C10—H10	119.9
C5—C6—H6	119.8	C10—C11—H11	119.9
C5—C6—C1	120.36 (12)	C12—C11—C10	120.15 (15)
C1—C6—H6	119.8	C12—C11—H11	119.9
C6—C1—C7	120.58 (12)	C11—C12—H12	120.0
C2—C1—C6	118.97 (12)	C13—C12—C11	119.97 (14)
C2—C1—C7	120.44 (12)	C13—C12—H12	120.0
C1—C2—H2	119.4	C12—C13—H13	120.0
C1—C2—C3	121.23 (13)	C12—C13—C14	120.10 (15)
C3—C2—H2	119.4	C14—C13—H13	120.0
C4—C3—C2	119.21 (13)	C9—C14—C13	120.60 (14)
C4—C3—H3	120.4	C9—C14—H14	119.7
C2—C3—H3	120.4	C13—C14—H14	119.7

O3—C8—H8A	110.0	O2—C7—O1	122.78 (12)
O3—C8—H8B	110.0	O2—C7—C1	118.87 (12)
O3—C8—C9	108.51 (11)	O1—C7—C1	118.36 (12)
H8A—C8—H8B	108.4		
O3—C4—C5—C6	179.92 (14)	C2—C1—C7—O1	0.0 (2)
O3—C4—C3—C2	−179.61 (14)	C3—C4—C5—C6	0.5 (2)
O3—C8—C9—C10	45.48 (19)	C8—O3—C4—C5	172.07 (13)
O3—C8—C9—C14	−136.32 (14)	C8—O3—C4—C3	−8.5 (2)
C4—O3—C8—C9	−171.59 (12)	C8—C9—C10—C11	177.81 (14)
C4—C5—C6—C1	−0.2 (2)	C8—C9—C14—C13	−177.25 (14)
C5—C4—C3—C2	−0.2 (2)	C9—C10—C11—C12	−0.3 (3)
C5—C6—C1—C2	−0.4 (2)	C10—C9—C14—C13	1.0 (2)
C5—C6—C1—C7	178.37 (14)	C10—C11—C12—C13	0.4 (3)
C6—C1—C2—C3	0.7 (2)	C11—C12—C13—C14	0.2 (3)
C6—C1—C7—O2	0.9 (2)	C12—C13—C14—C9	−0.9 (2)
C6—C1—C7—O1	−178.79 (15)	C14—C9—C10—C11	−0.4 (2)
C1—C2—C3—C4	−0.4 (2)	C7—C1—C2—C3	−178.11 (14)
C2—C1—C7—O2	179.73 (14)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.82	1.81	2.6213 (15)	169

Symmetry code: (i) −*x*+2, −*y*, −*z*+1.