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ISSN 2414-3146

3-Methyl-2-(4-methylphenoxy)benzoic acid

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Received 12 May 2023
Accepted 7 July 2023

Edited by W. T. A. Harrison, University of Aberdeen, United Kingdom

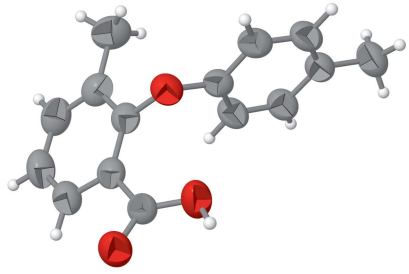
Keywords: crystal structure; carboxylic acid inversion dimer.

CCDC reference: 2280192

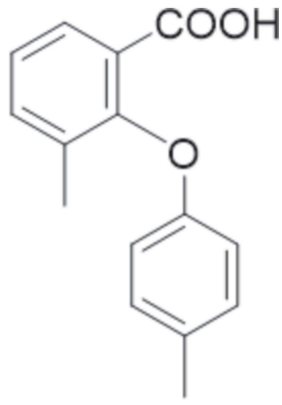
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{15}H_{14}O_3$, the dihedral angle between the aromatic rings is 86.7 (9)°. In the crystal, carboxylic acid inversion dimers linked by pairwise O—H···O hydrogen bonds are formed.

3D view



Chemical scheme



Structure description

2-Phenoxybenzoic acids are isosteres of anthranilic acids that are potential anti-inflammatory drugs and conformationally flexible molecules. Many anthranilic acids are polymorphic (Lopez-Mejias *et al.*, 2012; Sacchi, *et al.*, 2021). We wondered if 2-phenoxybenzoic acids would behave similarly to anthranilic acids in their polymorphism and as part of our work in this area, we now describe the synthesis and structure of the title compound, $C_{15}H_{14}O_3$.

There is one molecule in the asymmetric unit (Fig. 1). The molecule has a nearly perpendicular conformation as evidenced by the dihedral angle between the benzoic acid ring and the phenol ring [86.7 (9)°]. In the crystal, the molecules form carboxylic acid inversion dimers through pairwise O—H···O hydrogen bonds (Table 1, Fig. 2). Two weak C—H···π interactions are also observed.

Synthesis and crystallization

The title compound was synthesized through an Ullmann reaction between 2-chloro-3-methyl-benzoic acid and 4-methylphenol (Fig. 3) in an effort to investigate the effect of substitution position and pattern on the solid-state behavior of 2-phenoxybenzoic acids. A pure sample was dissolved in ethyl acetate at 60°C. Then, the solution was cooled to room temperature and was allowed to evaporate slowly in a fume hood. Colorless block-shaped crystals (Fig. 4) were harvested after a week.

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O3^{i}	0.86 (2)	1.80 (2)	2.6497 (14)	169 (3)
C4—H4···$Cg1^{ii}$	0.93	2.92	3.6557 (18)	137
C5—H5···$Cg2^{iii}$	0.93	2.97	3.8372 (15)	156

Symmetry codes: (i) $-x, -y+1, -z+1$; (ii) $-x, y+\frac{1}{2}, -z+\frac{3}{2}$; (iii) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$.

Refinement

Crystal and refinement data are listed in Table 2.

References

Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.

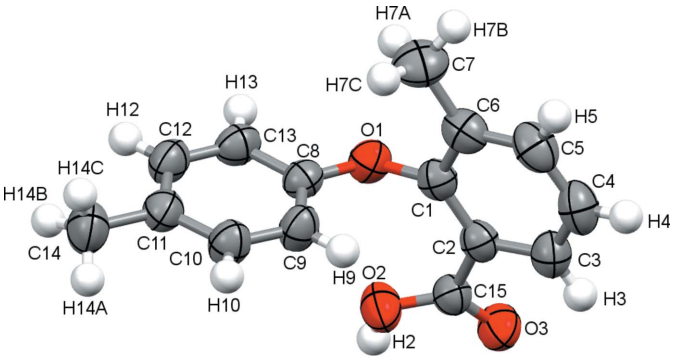


Figure 1
The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level.

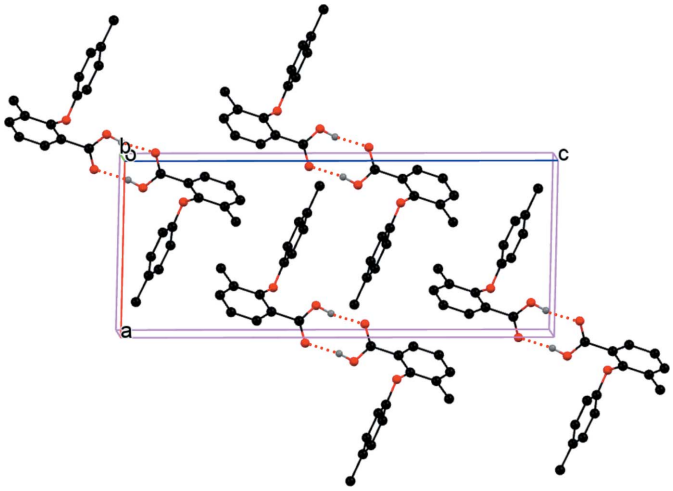


Figure 2
Packing of the molecules in the title compound.

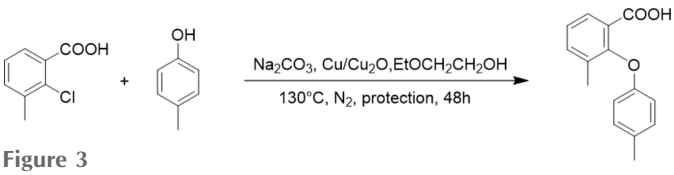


Figure 3
Reaction scheme.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{14}O_3$
M_r	242.26
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	293
a, b, c (Å)	8.95081 (17), 6.54110 (15), 21.9729 (4)
β (°)	91.2391 (18)
V (Å^3)	1286.17 (5)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.71
Crystal size (mm)	0.11 × 0.09 × 0.07
Data collection	
Diffractometer	Rigaku Oxford Diffraction, Synergy Custom system, HyPix
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2021)
T_{min}, T_{max}	0.856, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	7830, 2527, 2197
R_{int}	0.023
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.633
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.042, 0.124, 1.08
No. of reflections	2527
No. of parameters	169
No. of restraints	1
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.17, −0.17

Computer programs: *CrysAlis PRO* (Rigaku OD, 2021), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2018/3* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

López-Mejías, V., Kampf, J. W. & Matzger, A. J. (2012). *J. Am. Chem. Soc.* **134**, 9872–9875.

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Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.

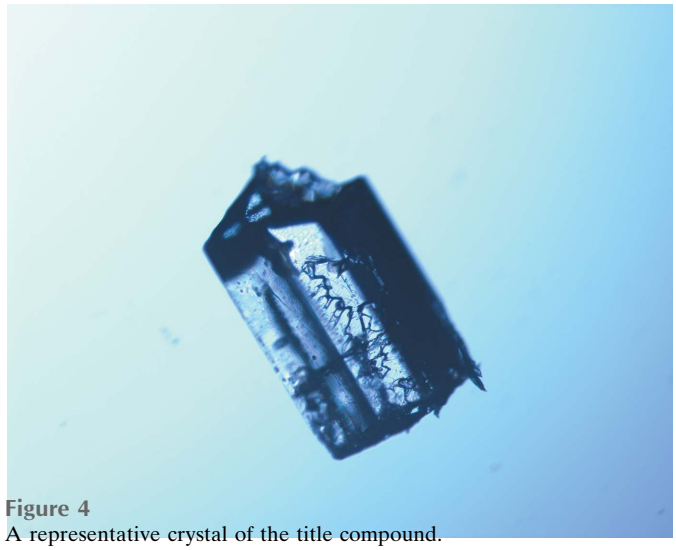

Figure 4
A representative crystal of the title compound.

full crystallographic data

IUCrData (2023). **8**, x230600 [https://doi.org/10.1107/S2414314623006004]

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Crystal data

$C_{15}H_{14}O_3$
M_r = 242.26
Monoclinic, $P2_1/c$
a = 8.95081 (17) Å
b = 6.54110 (15) Å
c = 21.9729 (4) Å
β = 91.2391 (18)°
V = 1286.17 (5) Å^3
Z = 4
$F(000)$ = 512
D_x = 1.251 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 5741 reflections
θ = 4.0–76.9°
μ = 0.71 mm^{-1}
T = 293 K
Block, colourless
0.11 × 0.09 × 0.07 mm

Data collection

Rigaku Oxford Diffraction, Synergy Custom system, HyPix diffractometer
Radiation source: Rotating-anode X-ray tube, Rigaku (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.0000 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysalisPro; Rigaku OD, 2021)
T_{min} = 0.856, T_{max} = 1.000
7830 measured reflections
2527 independent reflections
2197 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 77.6°, θ_{min} = 4.0°
h = −11→11
k = −8→8
l = −27→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.124
S = 1.08
2527 reflections
169 parameters
1 restraint
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0665P)^2 + 0.1182P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 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.

Refinement. The C-bound H atoms were placed geometrically (C—H = 0.93–0.96 Å) and refined as riding atoms.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.24620 (10)	0.17742 (14)	0.64109 (4)	0.0593 (3)
O2	0.15526 (12)	0.4152 (2)	0.54426 (5)	0.0769 (3)
H2	0.110 (3)	0.422 (5)	0.5094 (8)	0.190 (13)*
O3	−0.05029 (11)	0.58182 (18)	0.56942 (4)	0.0725 (3)
C1	0.21476 (13)	0.3529 (2)	0.67392 (6)	0.0536 (3)
C2	0.12444 (13)	0.5035 (2)	0.64797 (6)	0.0541 (3)
C3	0.08109 (16)	0.6693 (2)	0.68345 (7)	0.0656 (4)
H3	0.018988	0.769656	0.666817	0.079*
C4	0.13026 (18)	0.6842 (3)	0.74310 (7)	0.0745 (4)
H4	0.102602	0.795630	0.766625	0.089*
C5	0.22069 (17)	0.5335 (3)	0.76790 (6)	0.0728 (4)
H5	0.253775	0.546005	0.808144	0.087*
C6	0.26370 (15)	0.3642 (2)	0.73468 (6)	0.0639 (4)
C7	0.3572 (2)	0.1978 (3)	0.76307 (8)	0.0875 (5)
H7A	0.324401	0.067562	0.747699	0.131*
H7B	0.347063	0.200889	0.806463	0.131*
H7C	0.460117	0.218413	0.753209	0.131*
C8	0.38771 (14)	0.15863 (19)	0.61722 (6)	0.0531 (3)
C9	0.48515 (15)	0.3192 (2)	0.61186 (7)	0.0643 (4)
H9	0.458835	0.448913	0.625247	0.077*
C10	0.62304 (16)	0.2860 (2)	0.58630 (7)	0.0672 (4)
H10	0.688959	0.395109	0.582847	0.081*
C11	0.66564 (15)	0.0951 (2)	0.56573 (6)	0.0624 (3)
C12	0.56571 (17)	−0.0629 (2)	0.57189 (7)	0.0676 (4)
H12	0.591697	−0.192455	0.558302	0.081*
C13	0.42802 (17)	−0.0346 (2)	0.59764 (6)	0.0634 (4)
H13	0.362890	−0.144278	0.601829	0.076*
C14	0.81667 (19)	0.0625 (3)	0.53797 (9)	0.0852 (5)
H14A	0.844404	0.182635	0.515791	0.128*
H14B	0.811909	−0.052342	0.510806	0.128*
H14C	0.889706	0.036619	0.569672	0.128*
C15	0.07176 (14)	0.4993 (2)	0.58337 (6)	0.0554 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0555 (5)	0.0529 (5)	0.0694 (6)	−0.0043 (4)	−0.0001 (4)	−0.0034 (4)
O2	0.0720 (6)	0.1022 (9)	0.0567 (6)	0.0235 (6)	0.0036 (5)	−0.0043 (5)
O3	0.0633 (6)	0.0874 (8)	0.0666 (6)	0.0174 (5)	−0.0013 (4)	0.0021 (5)
C1	0.0477 (6)	0.0558 (7)	0.0573 (7)	−0.0064 (5)	0.0040 (5)	−0.0015 (5)
C2	0.0488 (6)	0.0583 (7)	0.0556 (6)	−0.0038 (5)	0.0058 (5)	−0.0010 (5)
C3	0.0608 (8)	0.0676 (9)	0.0689 (8)	0.0038 (6)	0.0092 (6)	−0.0065 (7)
C4	0.0707 (9)	0.0826 (11)	0.0707 (9)	−0.0025 (7)	0.0111 (7)	−0.0222 (8)
C5	0.0674 (8)	0.0947 (12)	0.0562 (7)	−0.0093 (8)	0.0003 (6)	−0.0114 (7)
C6	0.0559 (7)	0.0769 (9)	0.0587 (7)	−0.0078 (6)	−0.0021 (6)	0.0025 (6)

C7	0.0902 (11)	0.0955 (13)	0.0758 (10)	0.0046 (9)	−0.0197 (9)	0.0106 (9)
C8	0.0541 (6)	0.0514 (7)	0.0536 (6)	0.0022 (5)	−0.0039 (5)	0.0015 (5)
C9	0.0614 (7)	0.0487 (7)	0.0832 (9)	0.0010 (6)	0.0074 (6)	−0.0071 (6)
C10	0.0609 (8)	0.0582 (8)	0.0826 (9)	−0.0010 (6)	0.0072 (7)	−0.0027 (7)
C11	0.0651 (8)	0.0622 (8)	0.0599 (7)	0.0098 (6)	0.0012 (6)	−0.0003 (6)
C12	0.0807 (9)	0.0523 (8)	0.0698 (8)	0.0117 (7)	0.0016 (7)	−0.0061 (6)
C13	0.0735 (8)	0.0481 (7)	0.0683 (8)	−0.0023 (6)	−0.0031 (6)	−0.0007 (6)
C14	0.0781 (10)	0.0813 (11)	0.0969 (12)	0.0168 (9)	0.0168 (9)	−0.0063 (9)
C15	0.0523 (6)	0.0560 (7)	0.0581 (7)	0.0014 (5)	0.0052 (5)	0.0013 (5)

Geometric parameters (Å, º)

O1—C1	1.3875 (15)	C7—H7B	0.9600
O1—C8	1.3868 (15)	C7—H7C	0.9600
O2—H2	0.860 (10)	C8—C9	1.3719 (19)
O2—C15	1.2759 (16)	C8—C13	1.3857 (18)
O3—C15	1.2508 (16)	C9—H9	0.9300
C1—C2	1.3890 (19)	C9—C10	1.3841 (19)
C1—C6	1.3980 (18)	C10—H10	0.9300
C2—C3	1.3957 (19)	C10—C11	1.385 (2)
C2—C15	1.4862 (18)	C11—C12	1.375 (2)
C3—H3	0.9300	C11—C14	1.510 (2)
C3—C4	1.377 (2)	C12—H12	0.9300
C4—H4	0.9300	C12—C13	1.380 (2)
C4—C5	1.380 (2)	C13—H13	0.9300
C5—H5	0.9300	C14—H14A	0.9600
C5—C6	1.386 (2)	C14—H14B	0.9600
C6—C7	1.501 (2)	C14—H14C	0.9600
C7—H7A	0.9600		
C8—O1—C1	117.85 (9)	C9—C8—C13	120.10 (12)
C15—O2—H2	108 (2)	C13—C8—O1	116.38 (11)
O1—C1—C2	119.74 (11)	C8—C9—H9	120.4
O1—C1—C6	118.51 (12)	C8—C9—C10	119.27 (13)
C2—C1—C6	121.47 (12)	C10—C9—H9	120.4
C1—C2—C3	119.22 (12)	C9—C10—H10	119.1
C1—C2—C15	123.29 (12)	C9—C10—C11	121.88 (14)
C3—C2—C15	117.49 (12)	C11—C10—H10	119.1
C2—C3—H3	120.0	C10—C11—C14	120.94 (15)
C4—C3—C2	120.00 (14)	C12—C11—C10	117.47 (13)
C4—C3—H3	120.0	C12—C11—C14	121.59 (14)
C3—C4—H4	120.1	C11—C12—H12	119.0
C3—C4—C5	119.85 (14)	C11—C12—C13	121.92 (13)
C5—C4—H4	120.1	C13—C12—H12	119.0
C4—C5—H5	119.0	C8—C13—H13	120.3
C4—C5—C6	122.02 (13)	C12—C13—C8	119.36 (13)
C6—C5—H5	119.0	C12—C13—H13	120.3
C1—C6—C7	121.27 (14)	C11—C14—H14A	109.5

C5—C6—C1	117.42 (14)	C11—C14—H14B	109.5
C5—C6—C7	121.30 (14)	C11—C14—H14C	109.5
C6—C7—H7A	109.5	H14A—C14—H14B	109.5
C6—C7—H7B	109.5	H14A—C14—H14C	109.5
C6—C7—H7C	109.5	H14B—C14—H14C	109.5
H7A—C7—H7B	109.5	O2—C15—C2	118.22 (11)
H7A—C7—H7C	109.5	O3—C15—O2	122.84 (12)
H7B—C7—H7C	109.5	O3—C15—C2	118.92 (11)
C9—C8—O1	123.51 (11)		

O1—C1—C2—C3	173.51 (11)	C3—C4—C5—C6	−0.5 (2)
O1—C1—C2—C15	−7.17 (18)	C4—C5—C6—C1	1.5 (2)
O1—C1—C6—C5	−174.93 (12)	C4—C5—C6—C7	−177.62 (15)
O1—C1—C6—C7	4.16 (19)	C6—C1—C2—C3	−0.29 (19)
O1—C8—C9—C10	−178.73 (12)	C6—C1—C2—C15	179.03 (12)
O1—C8—C13—C12	178.25 (12)	C8—O1—C1—C2	105.42 (13)
C1—O1—C8—C9	−15.62 (18)	C8—O1—C1—C6	−80.60 (14)
C1—O1—C8—C13	165.09 (11)	C8—C9—C10—C11	0.2 (2)
C1—C2—C3—C4	1.3 (2)	C9—C8—C13—C12	−1.1 (2)
C1—C2—C15—O2	−30.72 (19)	C9—C10—C11—C12	−0.4 (2)
C1—C2—C15—O3	150.82 (13)	C9—C10—C11—C14	179.97 (14)
C2—C1—C6—C5	−1.06 (19)	C10—C11—C12—C13	−0.2 (2)
C2—C1—C6—C7	178.03 (13)	C11—C12—C13—C8	0.9 (2)
C2—C3—C4—C5	−0.9 (2)	C13—C8—C9—C10	0.5 (2)
C3—C2—C15—O2	148.61 (14)	C14—C11—C12—C13	179.47 (14)
C3—C2—C15—O3	−29.85 (18)	C15—C2—C3—C4	−178.07 (13)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O3^{i}	0.86 (2)	1.80 (2)	2.6497 (14)	169 (3)
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Symmetry codes: (i) −*x*, −*y*+1, −*z*+1; (ii) −*x*, *y*+1/2, −*z*+3/2; (iii) −*x*+1, *y*+1/2, −*z*+3/2.