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N-Benzyl-2-(2-chloro-4-methylphenoxy)-acetamide

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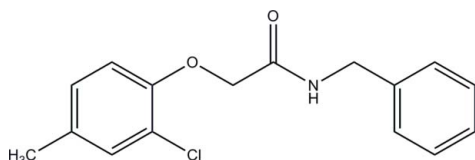
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 Key indicators: single-crystal X-ray study; $T = 273$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.046; wR factor = 0.133; data-to-parameter ratio = 17.8.

The structure determination of the title compound, $\text{C}_{16}\text{H}_{16}\text{ClNO}_2$, was performed as part of a project on the interactions between small organic molecules and proteins. In the crystal structure, the dihedral angle between the two aromatic rings is 16.14 (12)°. The molecules are connected via $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonding into chains, which extend in the direction of the b axis.



Experimental

Crystal data

 $\text{C}_{16}\text{H}_{16}\text{ClNO}_2$
 $M_r = 289.75$

 Orthorhombic, $Pbca$
 $a = 11.9900$ (18) Å

 $b = 9.2986$ (14) Å

 $c = 25.868$ (4) Å

 $V = 2884.1$ (7) Å³
 $Z = 8$

 Mo $K\alpha$ radiation

 $\mu = 0.27$ mm⁻¹
 $T = 273$ (2) K

 $0.15 \times 0.10 \times 0.10$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer

Absorption correction: none

15831 measured reflections

3286 independent reflections

 1964 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.051$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.045$
 $wR(F^2) = 0.133$
 $S = 0.96$

3286 reflections

185 parameters

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 0.21$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.22$ e Å⁻³
Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N}-\text{H}\cdots\text{O}2^i$	0.80 (2)	2.09 (3)	2.885 (2)	169 (2)

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2110).

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supporting information

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***N*-Benzyl-2-(2-chloro-4-methylphenoxy)acetamide**

Zhu-Bo Li, Hua Zuo, Wen-Liang Dong, Xiao-Yan He and Zhang-Bao Chen

S1. Experimental

A solution of 2-chloro-4-methylphenol (1.0 mmol), *N*-benzyl-2-chloroacetamide (1.1 mmol), K₂CO₃ (1.1 mmol) in CH₃CN (20 ml) was refluxed for 3 h. Afterwards the mixture has cooled down to room temperature the solvent was evaporated under reduced pressure. The residue was poured into water and adjusted the pH 6–7. The aqueous phase was extracted with ethyl acetate, washed with brine and dried over anhydrous MgSO₄. Finally the product was purified by column chromatography on silica gel. Crystals of (I) suitable for X-ray diffraction were obtained by cooling of a solution of the title compound in a mixture of ethylacetate and hexane.

S2. Refinement

All C-H atoms were placed in geometrically calculated positions and refined using a riding model with C—H = 0.97 Å (for CH₂ groups) and 0.96 Å (for CH₃ groups), their isotropic displacement parameters were set to 1.2 times (1.5 times for CH₃ groups) the equivalent displacement parameter of their parent atoms. The N-H H atom was freely refined.

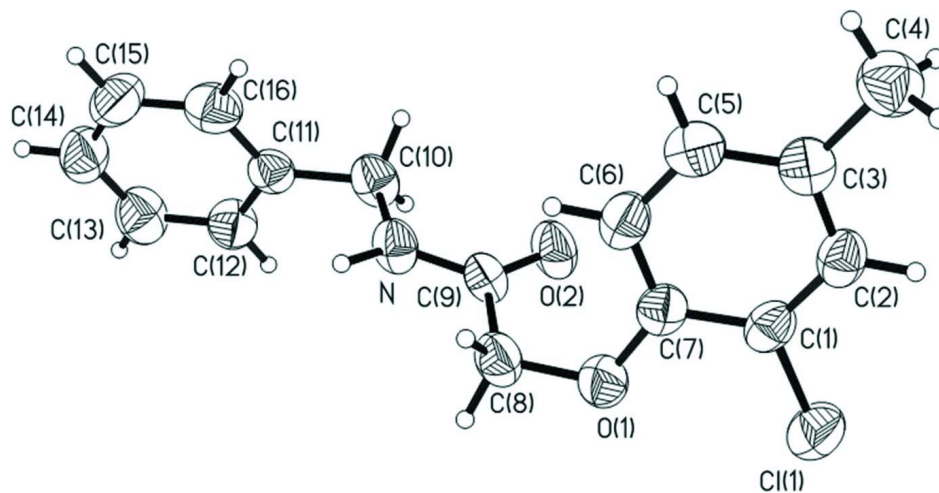


Figure 1

The molecular structure of (I) with labelling and displacement ellipsoids drawn at the 50% probability level.

***N*-Benzyl-2-(2-chloro-4-methylphenoxy)acetamide**

Crystal data

C₁₆H₁₆ClNO₂

M_r = 289.75

Orthorhombic, *Pbca*

a = 11.9900 (18) Å

b = 9.2986 (14) Å

c = 25.868 (4) Å

V = 2884.1 (7) Å³

Z = 8

$F(000) = 1216$
 $D_x = 1.335 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
 Cell parameters from 2043 reflections
 $\theta = 2.9\text{--}22.9^\circ$

$\mu = 0.27 \text{ mm}^{-1}$
 $T = 273 \text{ K}$
 Block, colorless
 $0.15 \times 0.10 \times 0.10 \text{ mm}$

Data collection

Bruker SMART CCD area-detector
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 φ and ω scans
 15831 measured reflections
 3286 independent reflections

1964 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.051$
 $\theta_{\text{max}} = 27.5^\circ$, $\theta_{\text{min}} = 1.6^\circ$
 $h = -15 \rightarrow 15$
 $k = -12 \rightarrow 11$
 $l = -25 \rightarrow 33$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.046$
 $wR(F^2) = 0.133$
 $S = 0.96$
 3286 reflections
 185 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 0.774P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.21 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.22 \text{ e \AA}^{-3}$

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N	0.25818 (17)	0.4733 (2)	0.38688 (7)	0.0475 (5)
H	0.2699 (18)	0.557 (3)	0.3929 (9)	0.049 (7)*
C11	0.03007 (5)	0.17559 (7)	0.57574 (3)	0.0657 (2)
O1	0.12962 (13)	0.38711 (16)	0.50994 (6)	0.0518 (4)
O2	0.19800 (14)	0.26379 (16)	0.42037 (6)	0.0557 (4)
C1	0.17217 (18)	0.2116 (2)	0.57204 (8)	0.0449 (5)
C2	0.24535 (19)	0.1359 (2)	0.60276 (8)	0.0479 (5)
H2	0.2178	0.0675	0.6257	0.057*
C3	0.35942 (19)	0.1599 (2)	0.60016 (9)	0.0484 (5)
C4	0.4390 (2)	0.0750 (3)	0.63326 (10)	0.0669 (7)
H4A	0.5140	0.1053	0.6263	0.100*
H4B	0.4315	-0.0255	0.6255	0.100*

H4C	0.4221	0.0911	0.6691	0.100*
C5	0.3965 (2)	0.2623 (3)	0.56529 (9)	0.0518 (6)
H5	0.4726	0.2803	0.5626	0.062*
C6	0.32366 (19)	0.3384 (2)	0.53445 (9)	0.0498 (6)
H6	0.3514	0.4062	0.5113	0.060*
C7	0.20977 (18)	0.3153 (2)	0.53747 (8)	0.0431 (5)
C8	0.1658 (2)	0.4792 (2)	0.46959 (8)	0.0510 (6)
H8A	0.2239	0.5422	0.4825	0.061*
H8B	0.1038	0.5387	0.4584	0.061*
C9	0.21020 (18)	0.3948 (2)	0.42362 (8)	0.0428 (5)
C10	0.2954 (2)	0.4080 (2)	0.33864 (8)	0.0510 (6)
H10A	0.2365	0.3473	0.3250	0.061*
H10B	0.3597	0.3477	0.3454	0.061*
C11	0.32561 (18)	0.5196 (2)	0.29900 (8)	0.0441 (5)
C12	0.2495 (2)	0.5609 (2)	0.26193 (9)	0.0547 (6)
H12	0.1787	0.5201	0.2619	0.066*
C13	0.2765 (2)	0.6617 (3)	0.22488 (10)	0.0653 (7)
H13	0.2241	0.6880	0.2001	0.078*
C14	0.3801 (2)	0.7230 (3)	0.22441 (10)	0.0655 (7)
H14	0.3985	0.7901	0.1991	0.079*
C15	0.4562 (2)	0.6855 (3)	0.26101 (12)	0.0686 (7)
H15	0.5263	0.7282	0.2611	0.082*
C16	0.4294 (2)	0.5832 (3)	0.29846 (10)	0.0608 (7)
H16	0.4820	0.5577	0.3232	0.073*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N	0.0699 (13)	0.0305 (10)	0.0421 (10)	−0.0025 (9)	0.0022 (9)	−0.0059 (8)
C11	0.0507 (4)	0.0739 (5)	0.0724 (5)	−0.0075 (3)	0.0088 (3)	0.0085 (3)
O1	0.0589 (10)	0.0498 (9)	0.0468 (9)	0.0051 (8)	0.0059 (7)	0.0039 (7)
O2	0.0827 (12)	0.0299 (8)	0.0546 (10)	0.0008 (8)	0.0042 (8)	−0.0021 (7)
C1	0.0479 (12)	0.0466 (12)	0.0403 (12)	−0.0045 (10)	0.0084 (10)	−0.0056 (10)
C2	0.0601 (14)	0.0441 (12)	0.0393 (11)	−0.0043 (11)	0.0071 (11)	−0.0013 (10)
C3	0.0573 (14)	0.0465 (13)	0.0413 (12)	0.0001 (11)	0.0005 (10)	−0.0075 (10)
C4	0.0665 (16)	0.0714 (17)	0.0627 (16)	0.0036 (14)	−0.0056 (13)	0.0011 (14)
C5	0.0480 (13)	0.0502 (14)	0.0572 (14)	−0.0033 (11)	0.0017 (11)	−0.0088 (11)
C6	0.0588 (14)	0.0441 (12)	0.0465 (13)	−0.0063 (11)	0.0104 (11)	−0.0021 (10)
C7	0.0525 (13)	0.0409 (11)	0.0360 (11)	−0.0006 (10)	0.0058 (9)	−0.0072 (9)
C8	0.0690 (15)	0.0373 (11)	0.0468 (13)	0.0048 (11)	0.0035 (11)	0.0006 (10)
C9	0.0546 (13)	0.0341 (11)	0.0397 (12)	0.0046 (9)	−0.0050 (10)	0.0012 (9)
C10	0.0690 (15)	0.0424 (12)	0.0414 (12)	0.0060 (11)	−0.0006 (11)	−0.0038 (10)
C11	0.0501 (13)	0.0410 (11)	0.0410 (12)	0.0054 (10)	0.0032 (10)	−0.0050 (9)
C12	0.0540 (14)	0.0569 (14)	0.0531 (14)	−0.0011 (12)	−0.0045 (11)	0.0017 (11)
C13	0.0795 (19)	0.0671 (16)	0.0494 (14)	0.0046 (14)	−0.0054 (13)	0.0077 (13)
C14	0.084 (2)	0.0584 (16)	0.0541 (16)	0.0035 (14)	0.0201 (15)	0.0046 (12)
C15	0.0574 (16)	0.0654 (17)	0.083 (2)	−0.0106 (13)	0.0191 (14)	−0.0030 (15)
C16	0.0523 (14)	0.0636 (16)	0.0665 (17)	0.0048 (12)	−0.0075 (12)	−0.0037 (13)

Geometric parameters (Å, °)

N—C9	1.329 (3)	C6—H6	0.9300
N—C10	1.458 (3)	C8—C9	1.521 (3)
N—H	0.80 (2)	C8—H8A	0.9700
C11—C1	1.739 (2)	C8—H8B	0.9700
O1—C7	1.370 (3)	C10—C11	1.503 (3)
O1—C8	1.418 (3)	C10—H10A	0.9700
O2—C9	1.230 (2)	C10—H10B	0.9700
C1—C2	1.377 (3)	C11—C16	1.378 (3)
C1—C7	1.390 (3)	C11—C12	1.378 (3)
C2—C3	1.387 (3)	C12—C13	1.380 (3)
C2—H2	0.9300	C12—H12	0.9300
C3—C5	1.385 (3)	C13—C14	1.366 (4)
C3—C4	1.505 (3)	C13—H13	0.9300
C4—H4A	0.9600	C14—C15	1.361 (4)
C4—H4B	0.9600	C14—H14	0.9300
C4—H4C	0.9600	C15—C16	1.395 (4)
C5—C6	1.378 (3)	C15—H15	0.9300
C5—H5	0.9300	C16—H16	0.9300
C6—C7	1.385 (3)		
C9—N—C10	121.07 (19)	O1—C8—H8B	109.3
C9—N—H	117.9 (16)	C9—C8—H8B	109.3
C10—N—H	120.9 (16)	H8A—C8—H8B	107.9
C7—O1—C8	117.56 (17)	O2—C9—N	123.1 (2)
C2—C1—C7	121.3 (2)	O2—C9—C8	121.51 (19)
C2—C1—C11	119.61 (17)	N—C9—C8	115.30 (18)
C7—C1—C11	119.12 (17)	N—C10—C11	111.74 (17)
C1—C2—C3	121.2 (2)	N—C10—H10A	109.3
C1—C2—H2	119.4	C11—C10—H10A	109.3
C3—C2—H2	119.4	N—C10—H10B	109.3
C2—C3—C5	117.3 (2)	C11—C10—H10B	109.3
C2—C3—C4	120.9 (2)	H10A—C10—H10B	107.9
C5—C3—C4	121.8 (2)	C16—C11—C12	118.1 (2)
C3—C4—H4A	109.5	C16—C11—C10	121.4 (2)
C3—C4—H4B	109.5	C12—C11—C10	120.5 (2)
H4A—C4—H4B	109.5	C11—C12—C13	121.1 (2)
C3—C4—H4C	109.5	C11—C12—H12	119.4
H4A—C4—H4C	109.5	C13—C12—H12	119.4
H4B—C4—H4C	109.5	C14—C13—C12	120.2 (3)
C6—C5—C3	121.8 (2)	C14—C13—H13	119.9
C6—C5—H5	119.1	C12—C13—H13	119.9
C3—C5—H5	119.1	C15—C14—C13	119.8 (2)
C5—C6—C7	120.8 (2)	C15—C14—H14	120.1
C5—C6—H6	119.6	C13—C14—H14	120.1
C7—C6—H6	119.6	C14—C15—C16	120.2 (2)
O1—C7—C6	125.9 (2)	C14—C15—H15	119.9

O1—C7—C1	116.42 (19)	C16—C15—H15	119.9
C6—C7—C1	117.6 (2)	C11—C16—C15	120.5 (2)
O1—C8—C9	111.79 (17)	C11—C16—H16	119.8
O1—C8—H8A	109.3	C15—C16—H16	119.8
C9—C8—H8A	109.3		
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C7—C1—C2—C3	-0.3 (3)	C10—N—C9—O2	2.8 (3)
C11—C1—C2—C3	178.82 (16)	C10—N—C9—C8	-174.4 (2)
C1—C2—C3—C5	-0.3 (3)	O1—C8—C9—O2	10.6 (3)
C1—C2—C3—C4	-179.0 (2)	O1—C8—C9—N	-172.11 (19)
C2—C3—C5—C6	0.3 (3)	C9—N—C10—C11	168.6 (2)
C4—C3—C5—C6	179.0 (2)	N—C10—C11—C16	83.7 (3)
C3—C5—C6—C7	0.3 (3)	N—C10—C11—C12	-96.4 (2)
C8—O1—C7—C6	9.4 (3)	C16—C11—C12—C13	0.8 (3)
C8—O1—C7—C1	-171.68 (17)	C10—C11—C12—C13	-179.1 (2)
C5—C6—C7—O1	178.02 (19)	C11—C12—C13—C14	-0.1 (4)
C5—C6—C7—C1	-0.9 (3)	C12—C13—C14—C15	-0.8 (4)
C2—C1—C7—O1	-178.11 (18)	C13—C14—C15—C16	1.0 (4)
C11—C1—C7—O1	2.7 (2)	C12—C11—C16—C15	-0.5 (3)
C2—C1—C7—C6	0.9 (3)	C10—C11—C16—C15	179.4 (2)
C11—C1—C7—C6	-178.22 (16)	C14—C15—C16—C11	-0.4 (4)
C7—O1—C8—C9	71.4 (2)		

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>
N—H...O2 ⁱ	0.80 (2)	2.09 (3)	2.885 (2)	169 (2)

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