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(*E*)-3-(3-Chlorophenyl)-*N*-(4-hydroxy-3-methoxybenzyl)acrylamide

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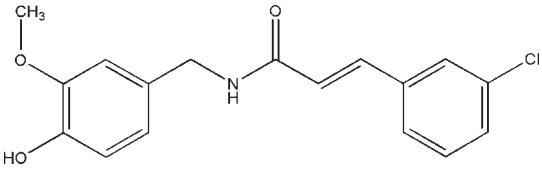
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; R factor = 0.036; wR factor = 0.083; data-to-parameter ratio = 14.2.

In the title compound, $C_{17}H_{16}ClNO_3$, the 4-hydroxy-3-methoxybenzyl group is planar [maximum atomic deviation = 0.0138 (16) Å] and is nearly perpendicular to the chlorobenzene ring, making a dihedral angle of 84.67 (4)°. The chlorobenzene and amide groups are located on the opposite sides of the C=C bond, showing an *E* configuration. The relatively long C=O bond distance of 1.2364 (19) Å and the short C—N bond distance of 1.341 (2) Å suggest electron delocalization in the amide fragment. Intermolecular O—H···O, N—H···O and weak C—H···O hydrogen bonding is present in the crystal structure.

Related literature

The title compound is a derivative of capsaicin. For the biological activity of capsaicin, see: Kaga *et al.* (1989). For a related structure, see: Huang *et al.* (2010). For electron delocalization in amide groups, see: Xia *et al.* (2009).



Experimental

Crystal data

$C_{17}H_{16}ClNO_3$ M_r = 317.76
Monoclinic, $P2_1/c$
a = 9.036 (3) Å
b = 14.972 (5) Å
c = 11.768 (4) Å
β = 95.047 (5)°
V = 1585.9 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 294 K
0.40 × 0.38 × 0.36 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
7733 measured reflections
2848 independent reflections
1680 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.083
S = 0.87
2848 reflections
201 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*···O3^{i}	0.86	2.12	2.960 (2)	165
O3—H3*A*···O1^{ii}	0.82	1.84	2.6491 (18)	172
C2—H2···O1^{iii}	0.93	2.41	3.298 (2)	161

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

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); 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: XU2783).

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supporting information

Acta Cryst. (2010). E**66**, o1700 [doi:10.1107/S1600536810022713]

(*E*)-3-(3-Chlorophenyl)-*N*-(4-hydroxy-3-methoxybenzyl)acrylamide

Liang-You Xia, Wen-Long Wang, Yan-Lan Huang and Shang Shan

S1. Comment

The title compound is a derivative of capsaicin, which has been shown a variety of biological activities including mutagenicity (Kaga *et al.* 1989). We prepared the compound recently in the laboratory and determined its crystal structure.

The molecular structure of the title compound is shown in Fig. 1. The chlorobenzene and amide groups are located on the opposite sides of the C7═C8 bond, showing the E molecular configuration. The hydroxymethoxybenzyl moiety is planar [the maximum atomic deviation being 0.0138 (16) Å for O2 atom], and is nearly perpendicular to the chlorobenzene ring with a dihedral angle of 84.67 (4)°. The dihedral angle between the amide fragment and hydroxymethoxybenzene ring is 89.69 (13)°, which agrees with 85.66 (9)° found in the related derivative of capsaicin, *N*-(4-hydroxy-3-methoxybenzyl)-3-chloro-2,2-dimethylpropanamide (Huang *et al.* 2010). The longer C9═O1 bond distance of 1.2364 (19) Å and the shorter C9—N1 bond distance of 1.341 (2) Å suggest the electron delocalization in the amide fragment, which is comparable to that found in the related compound *N*-(4-Hydroxy-3-methoxybenzyl)benzamide (Xia *et al.* 2009).

Intermolecular O—H···O, N—H···O and weak C—H···O hydrogen bonding are present in the crystal structure (Table 1), which helps to stabilize the crystal structure.

S2. Experimental

4-Hydroxy-3-methoxy benzylamine HCl salt (4.7 g, 25 mmol) and dimethylformamide (25 ml) were added to a 100 ml 3-necked flask equipped with an additional funnel, a thermometer and a magnetic stirrer. Water solution (10 ml) of NaOH (2.0 g) was added at room temperature. The mixture was stirred at 308 K for 30 min and then cooled to 273 K. An ether solution (10 ml) of 3-(3-chlorophenyl)acryloyl chloride (5.0 g, 25 mmol) was added dropwise at about 273 K over 20 min. After stirred for 2 h at room temperature the mixture was poured into water, and then extracted with ethyl acetate. The ethyl acetate extract was washed with 1 *M* HCl followed by saturated $NaHCO_3$ and brine. The extract was then dried over anhydrous Na_2SO_4 and filtered. Solvents were removed under vacuum at about 308 K to give a solid crude. Recrystallization was performed twice with an absolute ethyl acetate to obtain single crystals of the title compound.

S3. Refinement

H atoms were placed in calculated positions with O—H = 0.82, N—H = 0.86 Å, C—H = 0.93–0.97 Å, and refined in riding mode with U_{iso}(H) = 1.5U_{eq}(C) for methyl, 1.5U_{eq}(O) for hydroxy and 1.2U_{eq}(C,*N*) for the others.

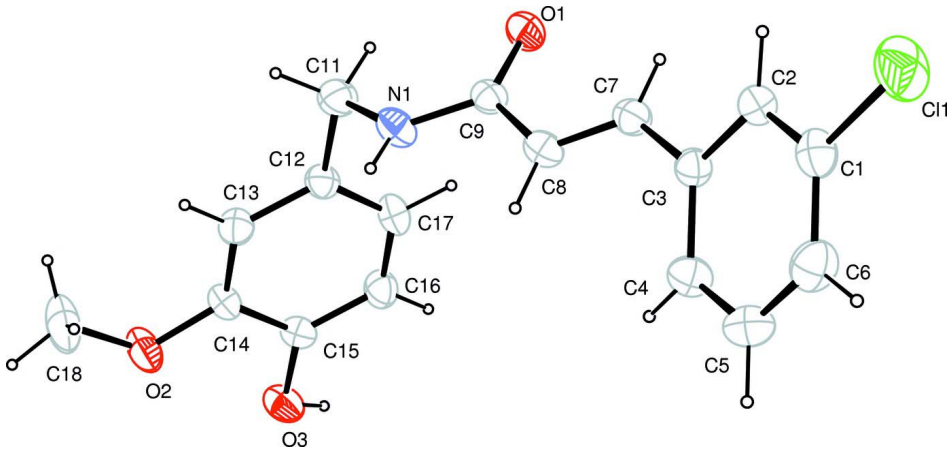

Figure 1

The molecular structure of the title compound with 30% probability displacement (arbitrary spheres for H atoms).

(*E*)-3-(3-Chlorophenyl)-*N*-(4-hydroxy-3-methoxybenzyl)acrylamide

Crystal data

$C_{17}H_{16}ClNO_3$
$M_r = 317.76$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 9.036$ (3) Å
$b = 14.972$ (5) Å
$c = 11.768$ (4) Å
$\beta = 95.047$ (5)°
$V = 1585.9$ (9) Å^3
$Z = 4$
$F(000) = 664$
$D_x = 1.331$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2246 reflections
$\theta = 2.6$–$24.8°$
$\mu = 0.25$ mm^{-1}
$T = 294$ K
Prism, colorless
0.40 × 0.38 × 0.36 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 10.0 pixels mm^{-1}
ω scans
7733 measured reflections
2848 independent reflections
1680 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 25.2°$, $\theta_{min} = 3.2°$
$h = -10 \rightarrow 10$
$k = -14 \rightarrow 17$
$l = -14 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.083$
$S = 0.87$
2848 reflections
201 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0403P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	1.00757 (8)	−0.14493 (4)	0.27966 (5)	0.0860 (2)
N1	0.40981 (17)	0.25930 (9)	0.46714 (12)	0.0486 (4)
H1N	0.4401	0.2856	0.4085	0.058*
O1	0.43789 (14)	0.13850 (8)	0.58155 (9)	0.0512 (3)
O2	0.26985 (16)	0.62811 (8)	0.51708 (10)	0.0635 (4)
O3	0.44518 (16)	0.63463 (8)	0.70403 (9)	0.0565 (4)
H3A	0.4801	0.6304	0.7705	0.085*
C1	0.9795 (2)	−0.03509 (13)	0.32316 (15)	0.0521 (5)
C2	0.8564 (2)	−0.01564 (12)	0.37947 (14)	0.0466 (5)
H2	0.7909	−0.0607	0.3961	0.056*
C3	0.8309 (2)	0.07197 (12)	0.41124 (14)	0.0419 (4)
C4	0.9333 (2)	0.13733 (13)	0.38823 (14)	0.0514 (5)
H4	0.9179	0.1962	0.4097	0.062*
C5	1.0569 (2)	0.11562 (14)	0.33406 (16)	0.0600 (5)
H5	1.1254	0.1598	0.3201	0.072*
C6	1.0807 (2)	0.02909 (15)	0.30013 (16)	0.0605 (6)
H6	1.1637	0.0146	0.2624	0.073*
C7	0.6954 (2)	0.09312 (12)	0.46534 (13)	0.0434 (5)
H7	0.6586	0.0499	0.5121	0.052*
C8	0.6219 (2)	0.16872 (12)	0.45275 (13)	0.0431 (5)
H8	0.6588	0.2131	0.4077	0.052*
C9	0.4838 (2)	0.18648 (12)	0.50668 (13)	0.0391 (4)
C11	0.2816 (2)	0.29694 (12)	0.51673 (16)	0.0534 (5)
H11A	0.2495	0.2566	0.5742	0.064*
H11B	0.2003	0.3041	0.4579	0.064*
C12	0.32141 (19)	0.38656 (11)	0.57021 (14)	0.0424 (4)
C13	0.2720 (2)	0.46507 (12)	0.51713 (14)	0.0451 (5)
H13	0.2101	0.4622	0.4498	0.054*
C14	0.3129 (2)	0.54727 (11)	0.56242 (14)	0.0427 (5)
C15	0.4062 (2)	0.55182 (11)	0.66299 (13)	0.0414 (4)
C16	0.4549 (2)	0.47407 (13)	0.71542 (14)	0.0498 (5)
H16	0.5172	0.4766	0.7826	0.060*
C17	0.4126 (2)	0.39228 (12)	0.66993 (15)	0.0522 (5)
H17	0.4462	0.3403	0.7070	0.063*
C18	0.1775 (3)	0.62875 (14)	0.41375 (17)	0.0868 (8)

H18A	0.2262	0.5978	0.3560	0.130*
H18B	0.1584	0.6893	0.3901	0.130*
H18C	0.0853	0.5996	0.4249	0.130*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0954 (5)	0.0612 (4)	0.1017 (5)	0.0236 (3)	0.0111 (4)	−0.0185 (3)
N1	0.0612 (11)	0.0352 (9)	0.0511 (9)	0.0042 (8)	0.0150 (8)	0.0023 (7)
O1	0.0566 (9)	0.0501 (8)	0.0473 (7)	−0.0021 (7)	0.0067 (6)	0.0142 (6)
O2	0.0950 (11)	0.0383 (8)	0.0529 (8)	0.0033 (7)	−0.0171 (7)	0.0047 (6)
O3	0.0828 (10)	0.0437 (8)	0.0415 (7)	−0.0120 (7)	−0.0033 (7)	−0.0015 (6)
C1	0.0558 (14)	0.0497 (12)	0.0502 (11)	0.0095 (11)	0.0011 (10)	−0.0019 (9)
C2	0.0452 (12)	0.0418 (11)	0.0520 (11)	−0.0003 (9)	−0.0005 (9)	0.0042 (9)
C3	0.0423 (12)	0.0406 (11)	0.0421 (10)	0.0008 (9)	0.0004 (8)	0.0013 (8)
C4	0.0567 (13)	0.0445 (11)	0.0535 (11)	−0.0041 (11)	0.0076 (10)	−0.0025 (9)
C5	0.0569 (14)	0.0612 (15)	0.0630 (12)	−0.0134 (12)	0.0107 (11)	0.0010 (11)
C6	0.0534 (14)	0.0742 (16)	0.0553 (12)	0.0056 (13)	0.0118 (10)	0.0004 (11)
C7	0.0480 (12)	0.0400 (11)	0.0418 (10)	−0.0041 (10)	0.0020 (8)	0.0026 (8)
C8	0.0542 (13)	0.0345 (11)	0.0412 (9)	−0.0027 (9)	0.0072 (9)	−0.0011 (8)
C9	0.0475 (12)	0.0324 (10)	0.0367 (9)	−0.0034 (9)	−0.0001 (8)	−0.0055 (8)
C11	0.0503 (13)	0.0409 (11)	0.0695 (12)	0.0010 (10)	0.0076 (10)	−0.0041 (10)
C12	0.0408 (11)	0.0360 (11)	0.0516 (11)	0.0038 (9)	0.0095 (9)	−0.0010 (9)
C13	0.0469 (12)	0.0439 (11)	0.0440 (10)	0.0016 (10)	0.0008 (8)	−0.0040 (9)
C14	0.0523 (12)	0.0347 (11)	0.0412 (10)	0.0035 (9)	0.0052 (9)	0.0033 (8)
C15	0.0488 (12)	0.0387 (11)	0.0373 (9)	−0.0045 (9)	0.0079 (8)	−0.0020 (8)
C16	0.0551 (13)	0.0499 (12)	0.0428 (10)	0.0025 (10)	−0.0043 (9)	0.0024 (9)
C17	0.0586 (14)	0.0424 (12)	0.0550 (11)	0.0130 (10)	0.0025 (10)	0.0075 (9)
C18	0.124 (2)	0.0588 (14)	0.0691 (14)	0.0223 (15)	−0.0405 (14)	0.0010 (11)

Geometric parameters (Å, º)

Cl1—C1	1.747 (2)	C7—C8	1.314 (2)
N1—C9	1.341 (2)	C7—H7	0.9300
N1—C11	1.456 (2)	C8—C9	1.473 (3)
N1—H1N	0.8600	C8—H8	0.9300
O1—C9	1.2364 (19)	C11—C12	1.512 (2)
O2—C14	1.366 (2)	C11—H11A	0.9700
O2—C18	1.413 (2)	C11—H11B	0.9700
O3—C15	1.366 (2)	C12—C17	1.376 (2)
O3—H3A	0.8200	C12—C13	1.386 (2)
C1—C6	1.370 (3)	C13—C14	1.379 (2)
C1—C2	1.375 (3)	C13—H13	0.9300
C2—C3	1.389 (2)	C14—C15	1.393 (2)
C2—H2	0.9300	C15—C16	1.372 (2)
C3—C4	1.389 (2)	C16—C17	1.377 (2)
C3—C7	1.464 (3)	C16—H16	0.9300
C4—C5	1.373 (3)	C17—H17	0.9300

C4—H4	0.9300	C18—H18A	0.9600
C5—C6	1.378 (3)	C18—H18B	0.9600
C5—H5	0.9300	C18—H18C	0.9600
C6—H6	0.9300		

C9—N1—C11	124.46 (15)	N1—C9—C8	114.38 (16)
C9—N1—H1N	117.8	N1—C11—C12	110.01 (15)
C11—N1—H1N	117.8	N1—C11—H11A	109.7
C14—O2—C18	117.95 (14)	C12—C11—H11A	109.7
C15—O3—H3A	109.5	N1—C11—H11B	109.7
C6—C1—C2	121.96 (18)	C12—C11—H11B	109.7
C6—C1—Cl1	119.05 (17)	H11A—C11—H11B	108.2
C2—C1—Cl1	118.99 (16)	C17—C12—C13	118.45 (16)
C1—C2—C3	119.33 (18)	C17—C12—C11	120.86 (16)
C1—C2—H2	120.3	C13—C12—C11	120.64 (16)
C3—C2—H2	120.3	C14—C13—C12	121.19 (16)
C2—C3—C4	118.94 (17)	C14—C13—H13	119.4
C2—C3—C7	119.09 (17)	C12—C13—H13	119.4
C4—C3—C7	121.95 (17)	O2—C14—C13	125.63 (15)
C5—C4—C3	120.44 (18)	O2—C14—C15	114.78 (15)
C5—C4—H4	119.8	C13—C14—C15	119.59 (15)
C3—C4—H4	119.8	O3—C15—C16	123.29 (15)
C4—C5—C6	120.7 (2)	O3—C15—C14	117.58 (15)
C4—C5—H5	119.6	C16—C15—C14	119.13 (16)
C6—C5—H5	119.6	C15—C16—C17	120.86 (16)
C1—C6—C5	118.6 (2)	C15—C16—H16	119.6
C1—C6—H6	120.7	C17—C16—H16	119.6
C5—C6—H6	120.7	C12—C17—C16	120.77 (16)
C8—C7—C3	124.84 (17)	C12—C17—H17	119.6
C8—C7—H7	117.6	C16—C17—H17	119.6
C3—C7—H7	117.6	O2—C18—H18A	109.5
C7—C8—C9	123.09 (17)	O2—C18—H18B	109.5
C7—C8—H8	118.5	H18A—C18—H18B	109.5
C9—C8—H8	118.5	O2—C18—H18C	109.5
O1—C9—N1	122.10 (17)	H18A—C18—H18C	109.5
O1—C9—C8	123.52 (16)	H18B—C18—H18C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O3^{i}	0.86	2.12	2.960 (2)	165
O3—H3*A*···O1^{ii}	0.82	1.84	2.6491 (18)	172
C2—H2···O1^{iii}	0.93	2.41	3.298 (2)	161

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) −*x*+1, *y*+1/2, −*z*+3/2; (iii) −*x*+1, −*y*, −*z*+1.